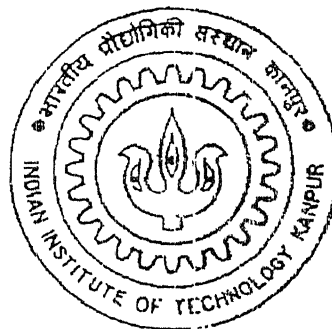# PRODUCTION OF MMC STRIPS USING SINGLE ROLL CONTINUOUS STRIP CASTER TECHNOLOGY

By

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PRODUCTION OF MMC STRIPS USING SINGLE ROLL CONTINUOUS STRIP CASTER TECHNOLOGY

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By

Mahendra H. Umaranikar

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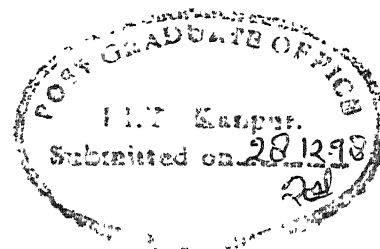INDIAN INSTITUTE OF TECHNOLOGY, KANPUR
December, 1998

CERTIFICATE

It is certified that the work contained in this thesis entitled *"Production of MMC Strips Using Single Roll Continuous Strip Caster Technology "*, by **Mahendra H. Umaranikar**, has been carried out under our supervision and that this work has not been submitted elsewhere for any degree.

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ABSTRACT

Composite materials are being used nowadays in every sphere of life. Among these composites, metal matrix composites (MMCs) with particulate reinforcement are finding applications in various fields The present investigation is two pronged First part consists of producing the MMC strips of aluminium with silicon carbide particulates as reinforcement using a single roll continuous strip caster (SRCSC), a technology developed in our laboratory by Mehrotra and coworkers for producing near-net-shape metal strips Various methods have been tried out for preparing the composite melt. Finally the vortex method is used. The wetting of particles by the melt is improved by alloying aluminium melt with magnesium, preheating of particles and stirring of the composite melt. In the second part the composite strips so produced are evaluated for their microstructure and mechanical properties Microstructural studies are carried out using optical as well as scanning electron microscope to examine the particulate distribution in the matrix, grain size and internal quality of the strips. Mechanical properties evaluation essentially included examination of tensile strength (ultimate tensile and 0.2% yield strength) and elongation, both in the longitudinal as well as transverse directions of cast strips. Also the effect of cold rolling, cold rolling and annealing, and hot rolling on the strip microstructure and mechanical properties has been studied in order to check the feasibility of this technology for commercial exploitation. Effect of process parameters such as the rotational speed of the caster drum, melt superheat, etc. on the microstructural and mechanical properties of cast composite strips has been investigated.

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Chapter 1

INTRODUCTION

It will not be an exaggeration to say that the composite materials have become the "Materials In Search of Problems". Indeed, you name the field and composites are present there – whether it is transportation, sports, leisure, medical, construction, etc. The composites are being employed from space applications to the kitchenware. The fiber reinforced polymers (**FRPs**), metal-matrix composites (**MMCs**), ceramic-matrix composites (**CMCs**), etc. are composites to name a few. Among these, the field of **MMCs** is developing at a rapid pace Several groups of investigators all over the world, are engaged in research on various aspects of the **MMCs** with an overall objective of improving their performance.

The **MMCs** possess high temperature hardness, higher strength, and higher modulus of elasticity than many of the conventional materials. They provide an alternative to the scarcely available materials being used currently in various fields. Metal-ceramic particle composites have been recognized as candidate materials for bearing applications because of their excellent wear resistance. These have a vast scope of application in the automobile and aerospace industries. The only drawback of the **MMCs** is that they possess less ductility than the matrix materials which is overshadowed by the above mentioned advantages.

Composites can be broadly classified into two categories:
  i.   Fiber reinforced composites, and
  ii. Particulate reinforced composites

This thesis is primarily concerned with the latter. The particulate reinforcement is, in most of cases, in the form of ceramic powders, e.g. alumina, silicon carbide, silica, graphite, etc.

1.1 METHODS OF PRODUCING MMCS

MMCs with ceramic particles as reinforcement can be prepared using several methods. These include both solid as well as liquid processing techniques. Powder metallurgy

(PM) method is the major route in solid-state processing through which MMCs are produced. Diffusion bonding has also been tried out successfully. However, solid state processing techniques require more time, costly equipments, and often these are cumbersome Compared to the solid state methods, liquid state processing has many advantages, e.g.

- secondary processes are minimized.
- time consumed is less.
- cost of production is lesser.
- intricate shapes can be produced.

Due to these advantages, liquid state processing techniques are becoming more popular. These include *(i) vortex method, (ii) pressure casting, (iii) squeeze casting, (iv) compocasting, (v) gas atomization method, (vi) vacuum casting*, [1] etc.

1.2 METHODS OF INCORPORATION OF THE PARTICLES

Each of the above mentioned methods employ a different methodology for incorporating the ceramic particles into the liquid metal matrix. For example in the vortex method [1], the ceramic particles are added in the metal when the matrix is in the liquid state. In this process a vortex is created by stirring the liquid metal and the reinforcing particles are incorporated in the vortex In the compocasting method [2], the reinforcement is done when the matrix is in the semi-solid state and then the mixture is vigorously stirred. In the pressure casting method [1], the particles are forced in the matrix using external pressure. In gas atomization technique [1], atomization of the liquid matrix is employed.

Liquid state processing techniques have their own demerits. In the compocasting method, a special stirrer capable of withstanding the corrosion and abrasion of the semi-solid melt is necessary. Also a more stringent temperature control is required to maintain the melt in semi-solid state during stirring. In pressure casting process, a source of compressed air is required. In gas atomization technique, a complicated set up for atomizing the melt is essential

The methods employed presently to produce strips of MMCs are described briefly in the next section

1.3 PRESENTLY EMPLOYED METHODS TO PRODUCE MMC STRIPS

This section presents an overview of the methods presently being used to produce MMC strips These include:

- Lamination
- Deposition
- Spray forming method
- P/M route
- Casting of MMC strips/sheets

These methods are described briefly in this section

1.3.1 Lamination

This is a group of methods in which, the strips are produced by laminating the components of the composite in alternate layers [3]. Temperature and pressure are applied to produce bonding at the interfaces. It starts with component materials in sheet, plate or powder form. These are then solid state bonded at the interface to produce the strips. The strips so produced are then passed through several processes such as sintering, rolling to get the final product. Surface preparation, pressure and time, chemical reaction between the component materials greatly influence the behaviour of the strips so produced. This group can be further subdivided into *adhesive bonding, melt bonding, infiltration bonding, reaction bonding, deformation bonding*, etc.

1.3.2 Deposition

It involves atomic or molecular scale transport of the component materials Chemical vapour deposition, electroplating, etc are a few processes of this type. These processes are too slow and are much costly for producing composite strips [4].

1.3.3 Spray Forming Method

In this method the matrix in the liquid form is sprayed onto the substrate. Here a gun is used in which a flame melts the matrix metal and also propels it in finely divided form towards the substrate. Very often the strips produced are complex having several spray formed layers. Al-SiC composite can be produced using this technique. The SiC particles are periodically injected into the atomized spray of liquid metal [3]. Here discrete interfaces do not exist between the metal and the reinforcement as is the case in bonded laminates.

1.3.4 Powder Metallurgy Method

In this method, the matrix metal is in the form of finely divided powder form. It is then compacted and consolidated using processes such as sintering, rolling, etc. MMCs using

metal aluminide as reinforcement are produced using the P\M route P\M route of processing can be further subdivided into the following types

1.3.4.1 ROLL COMPACTION: This method uses a specially designed rolling mill for the compaction of the powder. In the first step, a green strip of metal powder is produced using a binder and plasticizers. The strip is then passed through the rolling mill to obtain a coherent strip. Further processings like sintering, annealing, etc. are required to get the end product [4].

1.3.4.2 ISOSTASTIC PRESSING: External pressure equal in all directions (isostatic) is applied to the powdered mass to produce a coherent strip. This can be done in cold as well as hot condition In hot isostatic pressing (HIP), the powder is heated while being subjected to external pressure. The pressure source may be hydraulic or may be applied using gas.

A great amount of care is necessary in the P\M techniques to minimize the porosity

1.3.5 Casting of MMC Strips/Sheets

The conventional method of producing **MMC** strips/sheets involves several steps as listed below:

- Casting of ingots/billets
- Rolling of ingots/billets to produce strips
- Intermediate heat treatment
- Various finishing operations

One of the major problems in production of strips/sheets from composite ingots/billets comes during the rolling operation. Since the composites are more brittle than the metal matrix, rejection rate of rolled composite products due to crack formation is comparatively higher. Thus special precautions are necessary during the rolling operations making these quite expensive and energy extensive. The manpower required is also large. As a result, the total cost of production is high and the productivity is low.

Some of the commonly used methods of producing **MMC** castings have already been described about. Out of these the vortex method is perhaps the most convenient under the normal operating conditions. It does not require any complicated set up. Only a stirrer motor with graphite blades is necessary.

In this thesis, we propose to examine a new technique, involving liquid state processing, to produce **MMC** strips/sheets directly from melt using Single Roll Continuous

Strip Casting process This technique is essentially used for producing metal strips directly from the melt A detail description is given in appendix A

1.4   OBJECTIVE OF THE PRESENT INVESTIGATION

The presently employed method for production of strips/sheets of **MMCs** is through the processes described in the previous section. These methods call for more manpower and time for the secondary processes such as heat treatment, finishing, etc. As such these methods are cumbersome, costly and time consuming. These factors have to be minimized to have higher productivity. The present investigation is an attempt in this direction. The objective here is to explore the feasibility of extending the single roll continuous strip casting (SRCSC) method, developed in our laboratory for producing metal strips/sheets directly from the melt, for the production of **MMC** strips/sheets as well.

This investigation can be broadly divided into two parts The first part essentially deals with the production of **MMC** strips using SRCSC method Composites of aluminium with silicon carbide particles as reinforcement over a range of sizes are produced using vortex method. The **MMC** thus produced remains in the crucible and can be remelted in-situ. The melt so prepared is directly cast into strips/sheets using SRCSC, thus avoiding several passes of hot and/or cold rolling. The effect of process variables such as rotational speed of caster drum, superheat of the melt, etc on the strip thickness and quality are examined.

In the second part of this investigation the **MMC** strips/sheets produced in part one are evaluated for their microstructure and mechanical properties. Microstructure evaluation is primarily based on optical as well as scanning electron microscopy to examine: (i) the distribution of ceramic particles in the metal matrix, (ii) formation of precipitates, (iii) porosity distribution, etc. Mechanical properties evaluation involves tensile testing of as cast, cold rolled, cold rolled and annealed, and hot rolled strips.

This thesis essentially consists of five chapters. After this chapter on introduction, the second chapter covers the factors affecting mixing of ceramic particles into the metal matrix. The experimental procedure employed in this investigation is described in the third chapter. The results obtained are discussed in the forth chapter on results and discussion. Summary and conclusions of this investigation are presented in chapter five. An appendix in the end describes the single roll continuous strip caster (SRCSC) and its working briefly.

Chapter 2

MIXING OF PARTICLES IN THE MELT

From the point of view of the production and performance of the MMCs, it is of utmost importance that there should be a perfect mixing of particles in the matrix melt. A brief discussion of various aspects of particle mixing in metal matrix is presented in this chapter.

The mixing of reinforcing particles in metal matrix depends on the method of producing MMC as well as the process variables involved. The main factors which have a bearing on mixing behaviour are

1. **Interface stability**

2. **Physio-chemistry of wetting**

3. **Temperature and duration of preheat treatment of particles**

4. **Temperature of the matrix and holding time**

5. **Size of particles**

6. **Rate of addition of particles**

7. **Percentage of particles**

8. **Melt degassing**

These main factors are discussed below in some detail.

2.1 INTERFACE STABILITY

The possible interfaces in aluminium\aluminium-silicon alloy-particulate reinforcement systems can be between the aluminum and reinforcement, or between silicon and reinforcement, or between eutectic of aluminum-silicon and the reinforcement [6]. These possible interfaces are schematically shown in Fig. 2.1. In addition to these interfaces there are interfaces within the matrix region between the constituents of the matrix. For better mixing of reinforcement with the liquid metal it is necessary that there is be a stable interface between the matrix and the reinforcement. The interfacial interactions, method employed for incorporation of ceramic particles in the melt,the atmosphere during mixing, melt temperature, etc. affect the stability of the interface. These factors are dealt with in the following subsections.

2.1.1 Interfacial Interactions

The interfacial interactions can take place in various ways depending on the combination of matrix and the reinforcement, as discussed below.

2.1.1.1 DISSOLUTION: The dissolution of the reinforcing particles in the liquid melt may result in interfacial reaction. Driving force for this type of instability is the decrease in the surface area of the interface [6]. There is little or no change in the composition or the amount of the matrix. The principal disadvantage is the partial loss of the reinforcing material. Dissolution continues at higher temperatures and may be extensive if long duration of heating is necessary.

2.1.1.2 REACTION: For better compatibility, some degree of reaction between particles and the matrix is desirable. The extent of reaction should be sufficient to give enough strength to the metal-matrix interface. Excessive reaction may, however, lead to the consumption of particles, resulting in the weakening of the bond. This causes the failure of the MMC even at smaller loads. The kind of reaction that takes place depends on the melt temperature and the vulnerability of particles to the attack by the melt.

In the case of Aluminum-Mg alloy and alumina composite, the following reaction takes place at high temperature [5].

$$Al_2O_3 + Mg = MgAl_2O_3$$
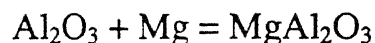
Magnesium spinel formed beyond a particular quantity has a negative impact on the interfacial stability. It increases the angle of contact between aluminum melt and the alumina particles by changing the interfacial energies. Therefore the excessive reactions should be avoided.

In the reactive composite systems e g.aluminium and SiC, the reaction taking place at the matrix-reinforcement interface is

$$4Al + 3SiC = Al_4C_3$$

Continued reaction forming a new compound at the interface is generally more detrimental to the properties of the composites A reaction may eat up the reinforcing particles thereby reducing the very purpose of reinforcement. In most such cases, the strength of the reactive zone is lower than that of the reinforcement. The cracks formed in the reactive zone determine the subsequent mechanical behaviour of the composite. The theory of brittle interface advanced by Metcalfe requires that the interfacial layer remains below a certain thickness to preserve strengthening [7].

2.1.1.3 EXCHANGE REACTION: When particles are in compound form e.g oxides some exchange reactions may take place, thereby reducing the compatibility of the matrix-particles. Due to this, one or more of the matrix elements concentrate at the interface resulting in concentration gradient across the interface which prohibits perfect mixing of particles. Reaction between the reinforcement and the matrix that contains two or more elements may be considered to occur in two steps.. In the first step the reaction product contains all of the matrix elements combined with the reinforcement to form a compound. However, thermodynamic conditions generally require that one or more of the matrix components concentrate in the compound Hence, in the second step exchange of the elements between the compound and the matrix occurs as the system approaches the equilibrium.

2.1.2 Effect of Alloying Elements

If the matrix is an alloy, e g Al-Si alloy, the alloying elements form distinct interfaces with particles. These interfaces may be between α- Al and reinforcement, between primary silicon and reinforcement or between eutectic of Al-Si and reinforcement. The interface between the α -aluminum and the ceramic particles is known to be favoured as compared to the other interfaces mentioned above [7]. This interface improves the mechanical properties of the composite Presence of secondary alloying elements, like Fe, Mg, Sb, etc. in the alloy as impurities may lead to the formation of intermetallic compounds such as $FeAl_3$, Mg_2Si, etc resulting in additional weak interfaces which, in turn, adversely affect the mechanical properties of the MMCs

2.1.3 Effect of Coating

If the reinforcement particles are coated with surface active elements {Li, Mg, Cu, etc. to improve wetting characteristics), the coating elements themselves may react with the matrix elements and also with the particles and form new interfaces Fig 2.2. In case of nickel coating [9, 10] on the reinforcement and with the aluminum as the matrix metal, the nickel reacts strongly with aluminum to form Ni_3Al_3 This is a very stable inter-metallic compound, which stabilizes the interface. The same thing happens in the case of copper coated particles. Copper can remain intact and form interfaces with matrix as well as particles improving the wetting characteristics.

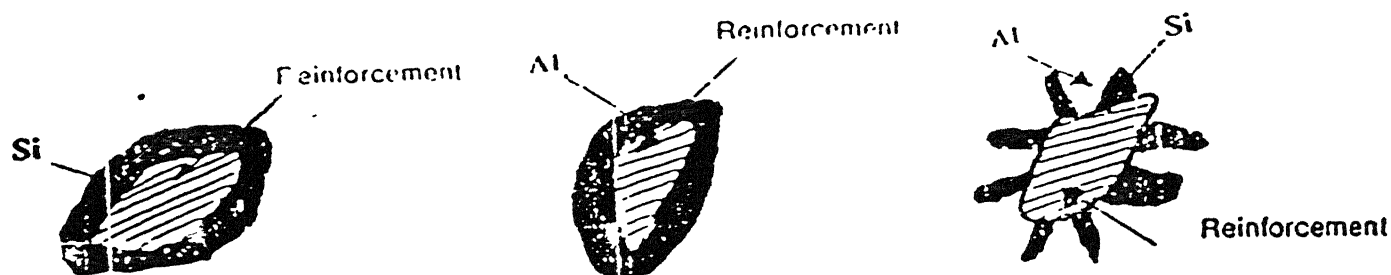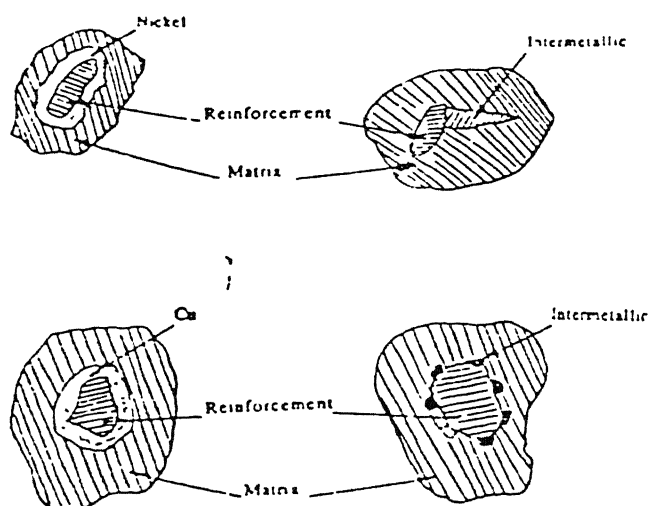**Fig 2.1 :** Schematic diagram showing possible interfaces in aluminium alloy composites [7]

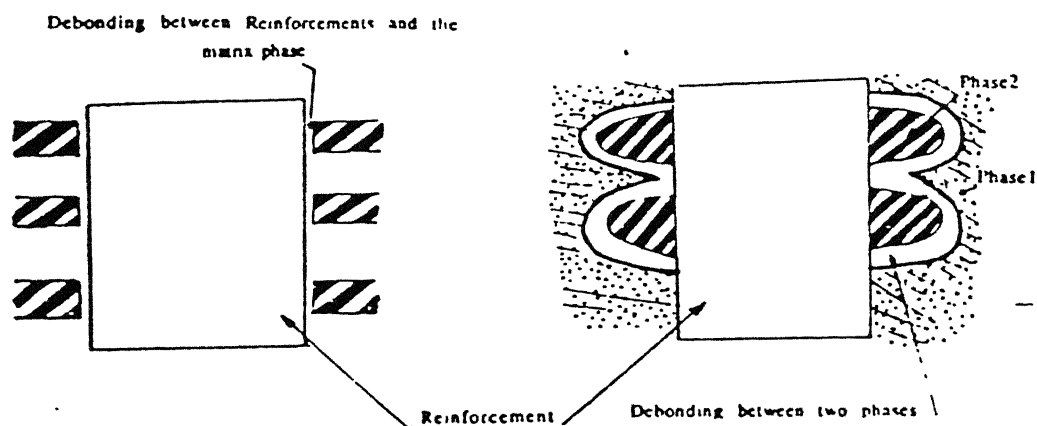**Fig. 2.2 :** Schematic diagram of interfaces with nickel or copper coated particles [7]

Fig. 2.3 : Schematic diagram showing debonding of interfaces [7]

2.1.4 Bonding Between Particles and Matrix Elements

Different interfaces have different surface energies and mechanical strengths depending upon the components forming it. The bonding between the reinforcing particles and the matrix elements determine the performance of the composite. Also the specific crystallographic planes of the reinforcement in contact with the specific crystallographic planes of the matrix affect the bonding. In case of aluminum-SiC interfaces, the dependence of the inter-facial binding energies on the crystal structures and crystallographic plane of SiC have been discussed by earlier investigators [8]. In case of aluminum–SiC interfaces, the bonding between aluminum and SiC particles can be between the layers of Si atoms or carbon atoms in SiC, and aluminum, and this further improves the mechanical properties.

If the bond between the matrix and the reinforcing particles is weak, the debonding is likely to take place at the interface between particles and the matrix. If the bonding between matrix and reinforcement is strong but between the matrix and other intermetallic compounds is weaker, then the debonding is likely to occur at the latter interface. this is represented schematically in Fig 2 3.

Thus the mixing of particles does not depend on the type of the matrix and particles used only, but also on the intermediate compounds that form during the mixing and their subsequent solidification behaviour Thus it is important to select the right combination of the matrix and the reinforcing particles to have proper mixing

2.1.5 Atmosphere During Mixing

The atmosphere maintained during mixing also plays an important role in preparation of sound MMCs If the mixing is done in air, the oxidation of metal takes place rapidly and to a higher extent due to high temperature. Metal oxides thus formed may react with the reinforcing particles and form some other compounds, which are not at all desirable in the final composites These compounds form interfaces with the reinforcement thereby reducing the strength and other properties. For example, Aluminum forms aluminum oxide in presence of air at a high temperature. If the reinforcement is silicon carbide, it reacts with magnesium present in the matrix along with aluminum and forms magnesium spinel. This spinel, if formed in excess, prevents good bonding between aluminum and silicon carbide particle. In case of other metals also the oxides formed play the similar role To prevent these effects, it is desirable to maintain inert atmosphere during mixing. The atmospheres of argon, nitrogen or mixture of these gases can be used depending on the combination of the matrix and the reinforcement.

It is apparent from the above discussion that several types of interfaces, some of which are highly complex in nature can be formed during the evolution of the microstructure in cast composites. The nature of these interfaces depends upon relative inter-facial energies, nucleation and growth of primary phases on reinforcements and on the solidification processing

2.2   PHYSIO-CHEMISTRY OF WETTING

For preparation of the MMCs, wetting of the reinforcing particles by the liquid metal plays a very crucial role. The wettability of a solid by a liquid melt is indicated by the contact angle θ. This angle is correlated to the three surface energies γ_{sg}, γ_{lg}, γ_{sl} of solid-gas, liquid-gas and solid-liquid interfaces Fig 2.4 respectively through the well known Young's equation

$$\gamma_{lg} \cos \theta = \gamma_{sg} - \gamma_{sl}$$
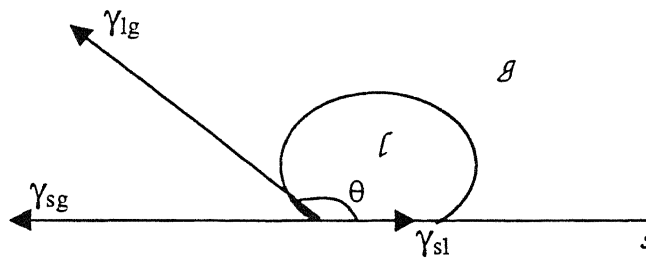
Fig. 2.4 : Schematic diagram of wetting principle

For wetting to take place, the necessary condition is

$$\cos \theta > 0$$
$$\text{i.e.} \quad \theta < 90°$$

From this condition it is inferred that lower the angle of contact, better is the wetting. In other words

$$\gamma_{sg} > \gamma_{sl}$$

The driving force F_w, for wetting can be defined as

$$F_w = \gamma_{sg} - \gamma_{sl}$$

In the extreme case when

$$F_w >= \gamma_{lg} \; ; \; \theta = 0°$$

the liquid spreads spontaneously on the solid For contact angle $\theta > 90°$, the capillarity effect requires an external pressure in order that the liquid wets the solid in contact with it. However, the application of pressure does not always completely solve the problem, since shrinkage during the solidification may be large enough to cause debonding or void formation

2.2.1 Factors Affecting Wettability

To analyse the phenomenon of wetting in detail, it is necessary to consider the factors that determine the extent of wetting. These are enumerated as follows:

- **Surface tension**

- **Adsorption**

- **Work of adhesion**

- **Presence of oxygen**

- **Surface characteristics of reinforcement**

2.2.1.1 SURFACE TENSION: It is the most important single factor determining the wetting characteristics. Higher surface tension (i.e. γ_{lg}) implies larger contact angle. As already stated above, for wetting the contact angle should be less than 90°. For metals with higher surface tension, it is therefore necessary that just sufficient chemical reaction takes place at the interface between the particle and the liquid metal so as γ_{sl} decreases the contact angle below 90° to ensure wetting to take place. It has been proven by Delannay and others [11] that a liquid wets a solid surface only if the energy of the bonds that are created across the interface exceeds the surface tension of the liquid. One of the ways of lowering the surface tension is by the addition of the alloying elements.

2.2.1.2 ADSORPTION: It is the physical phenomenon taking place between the liquid matrix atoms and the solid particles. If the matrix-reinforcement forms a reactive system, the adsorption is more thereby improving wetting. But again the reaction taking place should be less enough to avoid excessive damage to the particles.

2.2.1.3 WORK OF ADHESION: It is the measure of the strength of binding between two phases. In order to have a better wetting, work of adhesion should be equal to or greater than the surface tension of the liquid metal. For reactive metals, like aluminium, this is possible when there is chemical reaction between particles and the matrix. This effect is more pronounced for metals having higher affinity for oxygen [11].

2.2.1.4 PRESENCE OF OXYGEN: The interaction of the liquid metal with oxygen results either in formation of the oxides or alternatively yields a single metallic phase containing dissolved oxygen. Near the melting point, the metal forms its oxide. This oxide prevents the strong interface between metal and the reinforcement particles from being formed It also leads to higher angle of contact, which does not satisfy the basic wetting equation. Another point worth mentioning here is the substantial increase in the work of adhesion with the increasing concentration of oxygen in the melt. The phenomenon for wetting of sapphire by different metals is discussed by Mehrotra [12] In a wide range of oxygen partial pressures, γ_{sl} decreases roughly linearly with the logarithm of partial pressure of oxygen. Empirically, Mehrotra found that this slope increased with increasing free energy of formation of corresponding aluminates.

2.2.1.5 SURFACE CHARACTERISTICS OF PARTICLES: The nature of the surface of the reinforcing particles also affects wetting. If particles are oxidized or contaminated prior to the addition, wetting is affected with varying degrees. When the contamination is present, it changes the surface energies thereby changing the contact angle. Hence great care should be exercised while incorporating the particles into the metal In case of SiC - Aluminum, wetting experiments with oxidised SiC were performed [13] For this purpose SiC was oxidised at 1173 °K for about one hour. Table 2.1 shows the effect of this oxidation on contact angle. It is seen that for the same melt temperature and the holding time, the contact angle for oxidised SiC is much higher than that for the unoxidised SiC Good wetting can only be achieved at higher temperatures and higher holding times Thus it can be inferred that oxidation of SiC has a detrimental effect on the wetting characteristics of SiC particles in aluminum.

Melt temperature °K	Holding time Mm	Contact angle in degrees	
		Al/SiC	Al/oxidised SiC
973	15	120	133
	60	106	133
1003	15	100	130
	60	75	125
1043	15	70	121
	60	58	80

Table 2.1 : Contact angles for unoxidised and oxidised SiC with aluminium [13]

2.2.2 Approaches to Improve Wettability

As the wettability of reinforcing particles by the matrix melt is the most important factor in the preparation of the composites, it has been studied in detail. As there are limitations to the wetting properties in the as available conditions, attempts have been made to improve the wetting characteristics of the particles by employing various means These include:

i.   coating of reinforcing particles
ii. addition of alloying elements
iii. stirring
iv. pre-heating of ceramic particles

2.2.2.1 COATING OF REINFORCING PARTICLES: The wettability is highest in the case of mutual solubility or formation of intermatallic compounds. Coating of the reinforcing particles has been employed successfully by many researchers. Nickel coatings are especially used for aluminum composites. As already mentioned in section 2.1.4, in case of nickel coated alumina particles, a stable intermetallic compound is formed. This changes the interfacial energies, in a way so as to decrease the contact angle, thus, assisting better wetting between aluminium and alumina [10].

Silver, copper and chromium coatings have also been proposed. Silver coatings are conveniently formed by impregnation of the reinforcing network with a colloidal silver solution [14]. The high solubility of silver in aluminum provides good wetting without involving the formation of brittle compounds.

For aluminum composites, an alternate method is the sodium process [15]. This process consists of immersing the particles successively in liquid sodium at 550 °C, in a Sb-2% Mg bath at 600 °C and then in the aluminum melt Being highly electropositive, sodium wets alumina As sodium is soluble in tin and forms intermetallic compounds with tin, the sodium coating makes possible wetting by tin indirectly. In turn the tin-sodium coating promotes wetting by aluminum melt.

2.2.2.2 ADDITION OF ALLOYING ELEMENTS: Elements which have a high affinity for oxygen lower the interfacial energy of liquid metals with oxides. The most efficient alloying element reported to promote the wettability of the reinforcement is lithium. It is a highly electropositive element. Addition of the surface active elements like magnesium in aluminum melt, just prior to the particlulate addition, has also been employed in preparation of Al-graphite, Al-mica, Al-Al_2O_3 composites [7]. The wetting of SiC with aluminum can be improved by addition of silicon in the melt or by using alloy of Al -Si. It has been proved that the addition of 18% Si helps in wetting. This is due to the fact that Si reacts with aluminum to form Al_4C_3. This saves silicon in   SiC from reacting with aluminum thus maintaining its characteristics unchanged.

2.2.2.3 STIRRING: Stirring of the melt during and after addition of the reinforcing particles improves wettability of particles by the melt [2] In case of aluminum and silicon carbide

particles, stirring the melt for about 15 minutes after the addition of particles results in improved wetting.

2.2.2.4 PRE-HEATING OF PARTICLES: A pre-heat treatment of particles also improves wettability [16]. During the heat treatment, any oxides and contaminations present on the particles are removed. This directly affects the interfacial energies thereby assisting in improved wetting

2.3 TEMPERATURE AND DURATION OF PREHEAT TREATMENT

The reinforcing particles are heat treated before adding into the melt. This is done to prevent the apparent decrease in the viscosity of the melt Consequently it assists in the interfacial reactions between the matrix elements and the particles. The heat treatment is also necessary to avoid the agglomeration of the particles after the addition. It also removes moisture, if present any, and helps in removing contamination and oxide layers present on the particle surfaces.

In the vortex method used for the aluminum - alumina system, the alumina particles are preheated to a temperature of 900 °C [16]. The particles are held at this temperature for one hour. The temperature and the time of heat treatment are recommended after many trials Particles which are added to the matrix without being subjected to prior heat treatment are rejected by the matrix. The experimental observations made by Rohatgi et al [16] are represented in tabular form in Table 2.2. From this it is inferred that the optimum combination of preheating temperature and time is 900 °C and one hour. It applies to SiC particles and ellite clay as well The temperature and duration of heat treatment are different for different metal matrix-reinforcement systems.

2.4 PARTICLE SIZE

Recovery of the reinforcing particles in the solidified melt depends on the size and shape of the despersoids also [16]. From Table 2 2 it is seen that in case of alumina particle additions, the recovery is better with the addition of particles of 200 μ as compared with the recovery with particles of 60 μ . For silicon carbide particles the recovery is still less than that obtained with alumina. With the addition of less than 53 μ SiC particles, the recovery obtained is 33% only. It is, thus, inferred that for the particles of finer sizes, there is a greater probability of agglomeration, which prevents good mixing of particles into the matrix melt. In addition to the finer size, surface properties are also responsible for low recoveries. Hence for proper mixing of the particles, the size distribution should be optimum.

2.5 TEMPERATURE OF THE MELT AND HOLDING TIME

Temperature of the melt and the time for which it is held at this temperature during the preparation of the MMC, influence the wetting characteristics considerably. From

Particles	Wt % added	Preheat treatment		% recovery
		Temperature (°C)	Time (hr)	
200 μ Al_2O_3	3	300	24	Nil
200 μ Al_2O_3	3	500	15	Nil
200 μ Al_2O_3	3	700	4	Nil
200 μ Al_2O_3	3	900	1	96
200 μ Al_2O_3	3	900	0.5	70
60 μ Al_2O_3	3	900	1	83
Less than 53 μ illite clay	3	900	1	50
Less than 53 μ SiC	3	900	1	33
Less than 53 μ SiC	3	900	2	66

Table 2.2 : Influence of preheat treatment temperature and time on recovery of particles in Al-Si alloy [16]

the sessile drop experiments, it has been observed that wetting between the reinforcing particles and the aluminum/aluminum alloy melt is better at temperatures above 680 °C. In the experiments carried out by Rohatgi and co-workers [16], the temperature of melt, just prior to the introduction of particles, was maintained at 780 °C. Higher temperatures especially above 820 °C resulted in severe erosion of the stirrer blades. Also attempts to introduce particles below 700 °C led to rejection of the particles. Until the addition was completed care was taken to maintain the melt temperature above 740 °C. Experiments have been carried out to study the dependence of wetting - behaviour on the melt temperature and holding time for SiC-aluminium and Al_2O_3-aluminium systems [13]. For SiC-alumininium system, Fig. 2.5 shows variation of contact angle with melt holding time at 973 °K. It shows three distinct regions: first one with a sharp slope, second with a decreasing slope and the third one with almost straight portion From the above graph, it is seen that the wetting in this case is a time dependent phenomenon As can be seen from the figure, the contact angle is far more than 90° ~ 130° for the first 30 minutes. As the holding time is increased the contact angle goes on decreasing till it becomes almost constant after 90 minutes. The contact angle in this region is about 50°, which favours wetting. The variation of contact angle with the temperature at different holding times is also shown in Fig. 2. 6. It is seen that from 973 °K-1073 °K appreciable decrease in the contact angle is observed. The transition from non wetting to wetting is observed at 1013 °K. It is clear from the graphs that the transition temperature at which the non -wetting-wetting transition occurs decreases with increase in the holding time as well as with the increase in temperature.

Wetting of alumina with aluminum has also been investigated [13] Figure 2.7 shows the variation of contact angle with holding time for different temperatures. It also shows three regions as in case of SiC. At a temperature of 973 °K the contact angle decreases rapidly with time before reaching to a constant value after 15 minutes But the contact angle after stabilization is still greater than 90°. At 1003 °K there is no change in the angle with time because of the presence of an oxide layer around the particle. This layer prevents good wetting of the particle with the aluminum matrix. At higher temperatures this oxide layer is removed and wetting becomes easy at ~ 1273 °K.

2.6   RATE OF ADDITION OF PARTICLES

The rate of addition of particles also affects the mixing of particles in the melt. If higher rates are employed , the particles do not get enough time for perfect mixing. This results in poor wetting of the particles by the melt. This also results in the agglomeration of the particles and thereby very low recoveries Based on experimental investigations it has been shown that the rate of addition of the reinforcing particles into the matrix melt should be 30-40 g/min for good wetting and better recoveries [16].

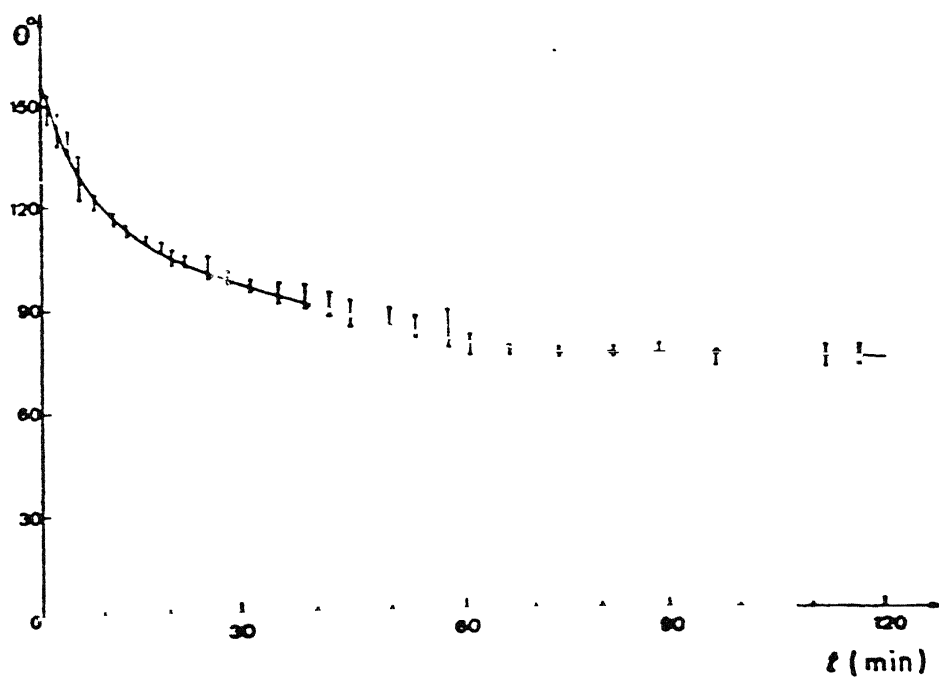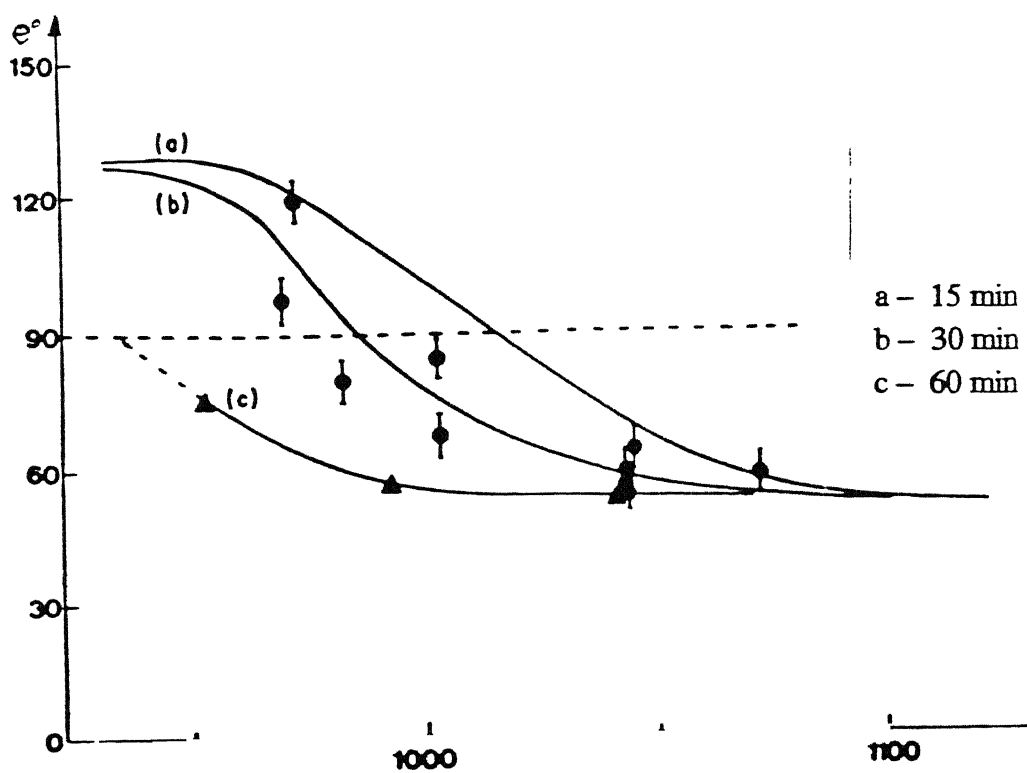**Fig. 2.5 : Variation of contact angle with time for Al/SiC system [13]**

Fig. 2.6 : Variation of contact angle with temperature at different time for Al/SiC system [13]
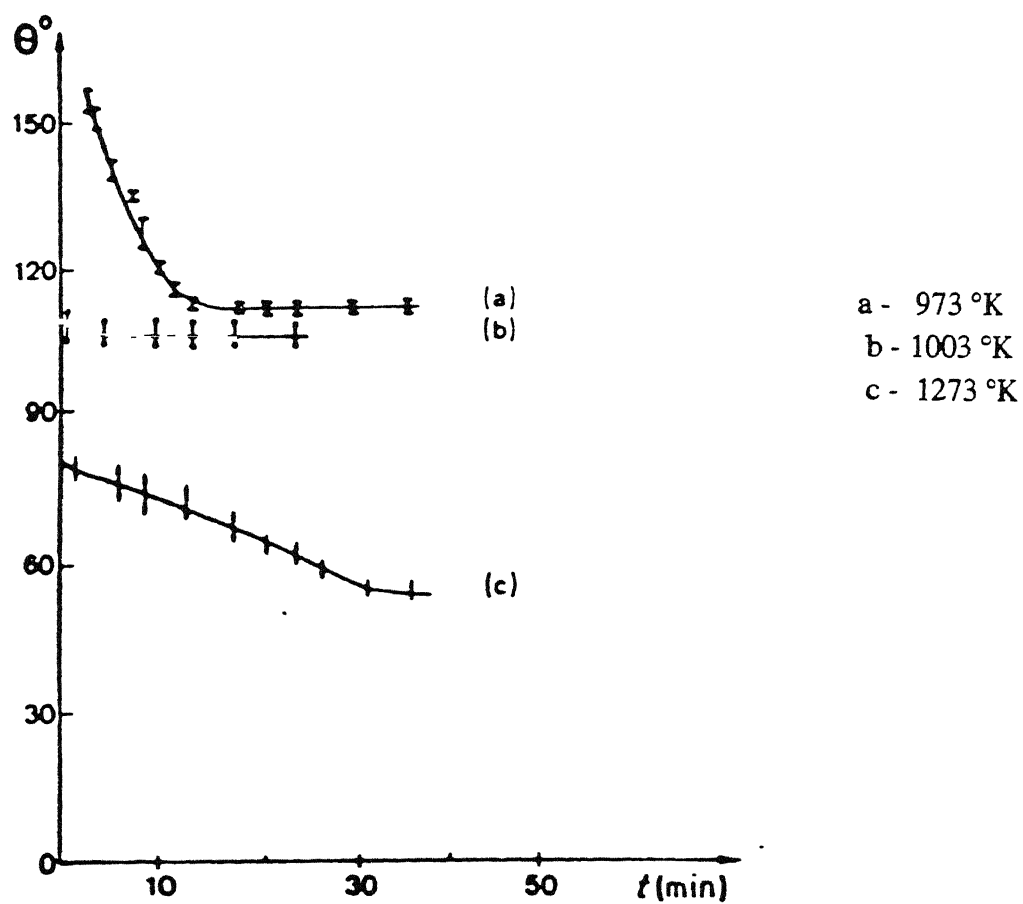
Fig. 2.7 : Variation of contact angle for Al/Al$_2$O$_3$ system [13]

2.7 PERCENTAGE OF PARTICLES

In the experimental investigations carried out by Rohatgi and Surofca [16], it was possible to introduce a maximum of only 6 wt% of alumina particles and 3 wt% of SiC particles, both of size 200 μ, in the aluminum melt. Due to local segregation, distribution of the particles in the melt was not even. The investigators inferred that the upper limits on particle containment could vary with the specific experimental conditions employed.

2.8 MELT DEGASSING

Sometimes it is necessary to degas the melt to remove dissolved gases. In case of aluminum alloys, degassing may be done by hexachloroethane or nitrogen gas prior to the addition of particles. Attempts to do so after addition of particles results in complete rejection of particles. This can be attributed to the reactions between aluminum and the chlorine present in the degassifier forming compounds at higher temperatures.

Each of the factors described in the previous sections influences the mixing of reinforcing particles in the metal matrix melt in its own way with varying severity. All these factors must be taken into consideration for ensuring a proper composite melt and necessary precautions must be taken to have higher recovery of particles.

Chapter 3

EXPERIMENTAL PROCEDURE

In this chapter, the experimental procedure employed in this investigation is described. First, the preparation of the composite melt is dealt with. This is followed by a brief description of the method of casting composite strips. The steps required for mechanical and microscopic testing of the strips so produced are discussed at the end.

3.1 PROCEDURE EMPLOYED FOR THE PREPARATION OF COMPOSITE MELT

Before employing the vortex method for the preparation of composite melt in this study, four other methods had been tried out. A brief description of these methods and the reasons for their failure are described in this section.

3.1.1   Particle Incorporation in Pure Aluminium Matrix

This was the first procedure attempted in which only commercial aluminium was used as the matrix material and no alloying element was added. Aluminium, however, contained some impurities like Fe, Si, Cu, etc. The metal was heated to above its melting point. After allowing time for stabilization, the melt was degassed with a mixture of hexachloroethane. The melt was stirred manually with graphite rod after addition of the degassifier for about two minutes. Any slag formed during this process was removed. At this stage SiC particles in the size range of -72 +120 mesh were added in the melt with a quantity of 6 wt %. No pre heat treatment was given to the particles. The rate of addition of the particles was maintained at 40 g/min. During addition of the particles, the melt was continuously stirred manually with the

graphite rod 15 minutes It was observed that even during stirring particles were floating onto the melt surface As soon as the stirring was stopped, almost the entire particle mass floated

3.1.2. Compocasting Method

As already described in the previous chapter, this is the casting process in which the melt is stirred vigorously in the semi-solid state Following this method, aluminium was heated to above its melting point It was then cooled at a controlled rate such that the melt became semi-solid (about 40% solid). At this stage stirring of the matrix was started using a motorised stainless steel stirrer at 400 RPM Simultaneously, the addition of ceramic particles was commenced Size distribution of particles and quantity were the same as in the previous case No pre heat treatment was given to the particles During the stirring care was taken to maintain the temperature of the furnace so as to keep the matrix in the semi-solid state. The stirring was done for half an hour, after that the matrix was allowed to cool down to room temperature.
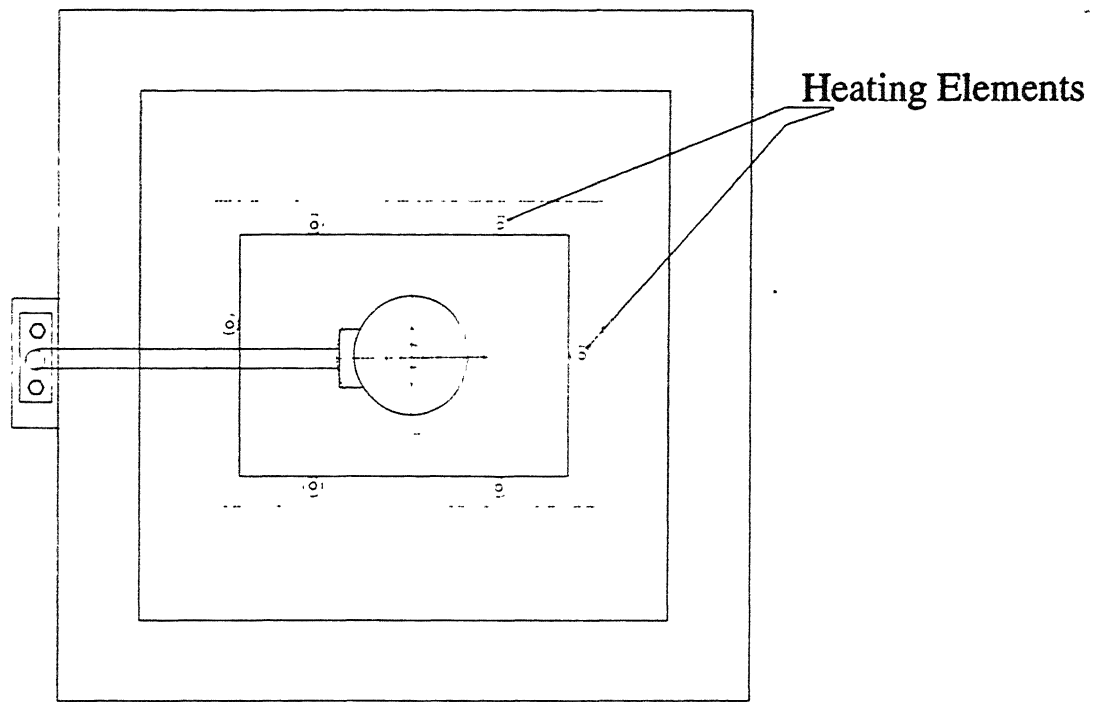
It was noted that, on remelting the composite mass, the particulate mass still floated onto the melt surface Severe erosion of stainless steel blades and shaft of the stirrer by the melt was also observed In 3-4 experimental runs the stirrer blades and the shaft were eroded to almost half of their original size

3.1.3 Vortex Method with Manual Stirring

In this procedure a vortex was created in the liquid melt with the help of manual stirring of the melt with graphite rod Untreated ceramic particles were added in the vortex. This method also proved unsuccessful with the result that the particles were rejected by the melt.

3.1.4 Vortex Method with Addition of Surface Active Alloying Agent and Heat-treated Particles

This process was found to be successful In this case the stirring was done with a motorised graphite stirrer. The ceramic particles were preheated at 900 °C for one hour. Alloying of aluminium with magnesium was done just prior to the addition of the particles. This method is discussed in detail below Figure 3 1 shows the experimental set up used for this method while Fig. 3 2 is a schematic diagram of the tundish used The parameters kept constant for all the experiments are given in Table 3 1 while Table 3.2 shows process variables used in the experiments Steps involved in the preparation of the melt are enlisted below
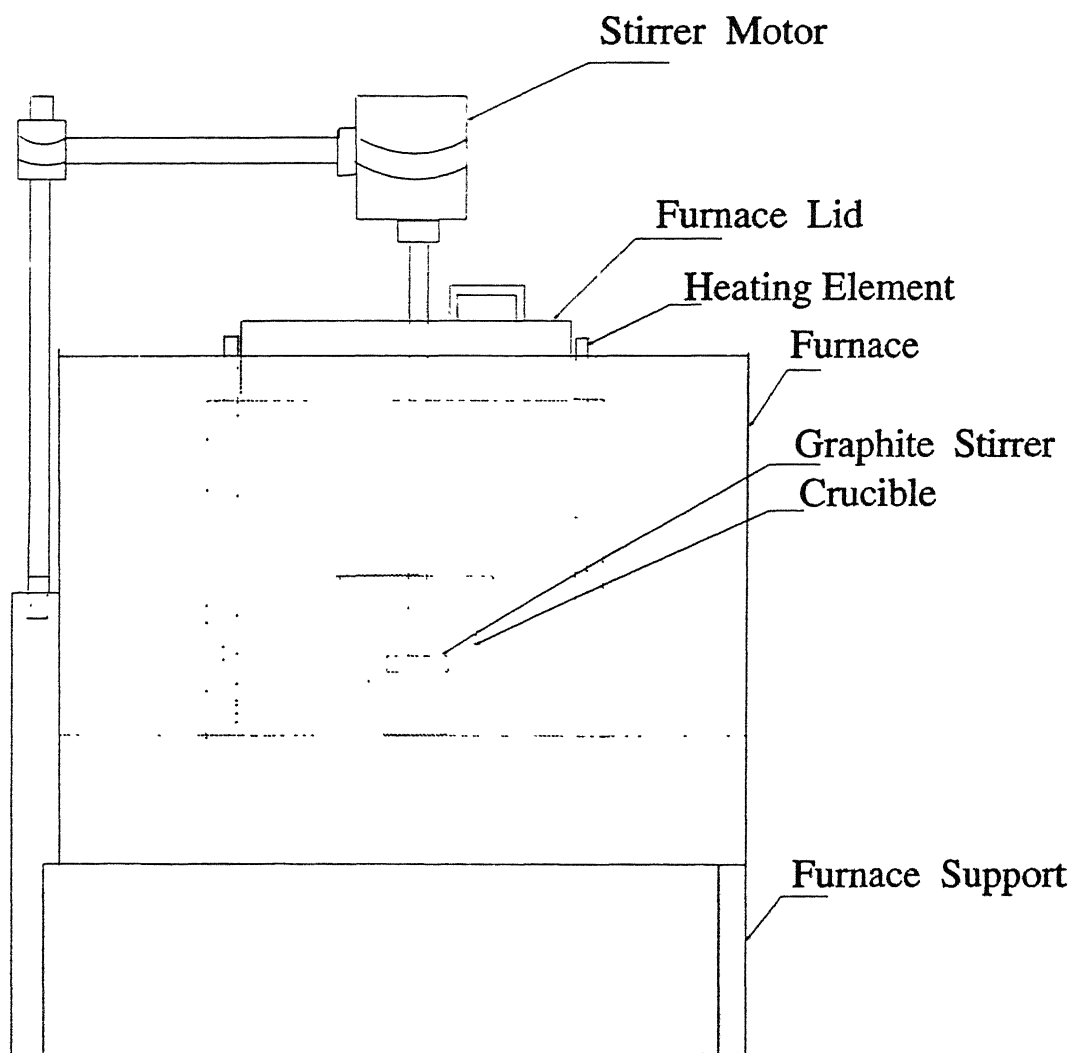
Fig. 3.1 : Schematic Diagram of Experimental Set-up
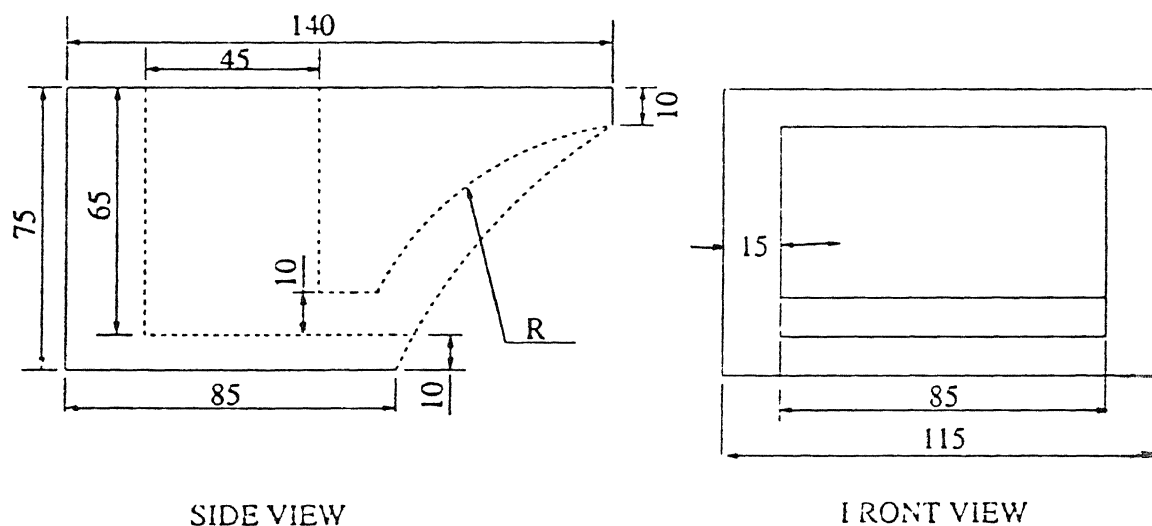
SIDE VIEW   FRONT VIEW

All DIMENSIONS ARE IN mm

Fig. 3.2 : Schematic diagram of tundish

Sr. No.	Variable	Range
1	Speed of rotation of Caster drum	12-26 RPM
2	Degree of superheat Of the melt	90 -120 °C
3	Height of liquid metal In the tundish	10-40 mm
4	Tundish nozzle gap	10-20 mm
5	Cooling water flow rate	0.4 GPM
6	Stand-off distance	10-20 mm

Table 3.1 : Process variables and their ranges

Quantity of C. P.	Particle Size	Pre-heating of C. P.	Quantity of Mg	Temp of Mg Addition	Temp Range of C. P. Addition	Rate of C. P. Addition	RPM of Stirrer	Duration of Stirring	Water Flow Rate
5 wt %	100 -200 μ	900 C for 1 Hour	0.5 wt %	720 C	740 C - 780 C	30 - 40 g/min	400	15 -20 min	0 4 GPM

(C. P. - Ceramic Particles)

Table 3.2 : Parameters Kept Constant for All the Experiments

♦ melting of aluminium

♦ addition of magnesium

♦ preheating of the particles

♦ incorporation of particles

♦ stirring

3.1.4.1 Melting of Aluminium: First the charge is heated above its melting point Care is taken to minimize the time required to attain the required temperature keeping in view the highly oxidizing characteristics of aluminium. The charge is melted in a resistance heating furnace in about four hours. When the temperature is reached to 720 °C, slag and dross, if any is removed. Now the melt is ready for the addition of magnesium.

3.1.4.2 Addition of Magnesium: Magnesium acts as a strong wetting agent in case of aluminium melt and ceramic particles used in these experiments. A specified quantity of magnesium (0 5 wt %) is added in the melt. Magnesium to be added, in the form of turnings, is wrapped in an aluminium pouch which is tied onto one end of the graphite rod with a thin aluminium wire. The graphite rod is then inserted into the melt, which is manually stirred with the same rod for about 5 minutes. Care is taken to minimize the heat losses during this whole process After some time, any slag, if present, is removed.

3.1.4.3 Preheating of the Particles: It is necessary to preheat the reinforcing particles prior to their incorporation into the melt. The preheating removes the coatings present, if any, on the surface of the particles. This aspect has been dealt with in detail in the literature review [16]. The particles are heated at 900 °C for one hour in a resistance heating furnace. The heating rates of both furnaces i. e. the furnace used for melting aluminium and the one used for preheating the particles are synchronised such that the required temperatures are reached almost simultaneously in both the furnaces. After one hour the ceramic particles are removed from the preheating furnace for incorporating them into the melt.

3.1.4.4 Incorporation of the Particles in the Melt: This is perhaps the most important step in the preparation of the melt for composites The time lag between removal of the ceramic particles from the preheating furnace and addition into the melt is kept to a minimum to avoid excessive cooling of the particles. This time lag should not exceed 10 minutes [16]. The

particles are added in the melt while the melt is being continuously stirred with the graphite stirrer from the top. Care is taken such that the particles are added into the vortex formed by the stirring action for better mixing. The rate of addition is maintained at 30-40 g/min for better recovery. The temperature of the melt is maintained between 740 °C-780 °C to avoid rejection of the particles

3.1.4.5 Stirring: For the stirring purpose, a stirrer motor of 1 hp is used. The speed of stirrer is kept constant at 400 RPM. Before starting the stirring, the blades and the stirrer shaft are preheated in the furnace to avoid thermal shocks and the consequent reduction in the melt temperature The position of the blades is adjusted such that they are at half the depth of the melt in the crucible. After addition of the reinforcing particles, stirring is continued for 15- 20 minutes to have higher recovery of the particles [16].

Temperature is continuously monitored during the whole process of melt preparation using a thermocouple The slag and any other impurities floating onto the melt, are removed. When temperature of the melt is reached to the desired superheat, it is ready for pouring.

3.2 CASTING OF COMPOSITE STRIP

Before pouring the melt into the tundish, it is ensured that the tundish is preheated to minimize thermal shocks. The caster is switched on at a particular speed of rotation using the stepper motor. A prespecified water flow is set for the cooling purpose. The composite melt from the crucible is poured manually into the tundish. Pouring is done in such a way that the melt level in the tundish reaches the marked level quickly enough and then remains at that level during the entire casting process. A melt pool is formed in the annular space between the rotating caster drum and the tundish wall. As soon as the drum comes in contact with the melt, formation of solid strip commences at the drum surface. This strip is gently withdrawn from the pool and brought over the drum surface with the help of dummy bar which is a bent aluminium strip of the same width as that of the strip. This strip is peeled off from the drum with a knife edge as the strip comes to the top of the drum and then it travels on its own over to the guiding platform A detail description of the caster is given in the appendix.

The casting process is continued as long as melt is poured from the crucible or the length of the guiding platform is reached. Then the strip is allowed to cool down in the ambient conditions. The operating parameters such as speed of rotation of caster drum, water flow rate, degree of superheat are noted and marked on the strip. The strip is now ready for preparing the samples for mechanical and microscopic examinations. This part is discussed in some detail in the following section.

## 3.3   SAMPLE PREPARATION FOR MECHANICAL/MICROSCOPIC TESTS

One of the features of this investigation is the microstructure and mechanical properties evaluation of the composite strips produced using the single roll continuous strip caster. The mechanical properties examination includes tensile strength and ductility. In micro-structure evaluation, distribution of the reinforcing particles and the overall microstructure of the strips are to be observed For this, samples are cut from the strips in the longitudinal as well as transverse direction It is ensured that these samples are flat, without any protrusions and cracks on the surface. If scales and protrusions are found on the strips, the former is scraped with a wire brush while the latter is removed by proper filing. For the specimens, small pieces with dimensions 100 mm x 15 mm are taken in the longitudinal and transverse directions. As the shearing may result in some changes in the mechanical properties, the pieces are cut with a power saw. The specimens in the longitudinal and transverse directions are prepared as per the ASTM and British standards respectively [18]. These standard dimensions are shown in Fig. 3.3. Width of the strips, limited by the length of the roll caster, is insufficient for preparing tensile test specimens in the transverse direction as per the ASTM standards For this reason British standards are used for preparing samples in the transverse direction for tensile strength testing.

The tests are made on the strips subjected to the following conditions.

- as cast
- cold rolled
- annealed
- hot rolled

3.3.1.1 AS-CAST SPECIMENS: At least two specimens are cut from the strip. No additional preparation is required for these specimens other than the removal of scales and protrusions. The samples are cut from different positions on the strip to avoid effects of agglomeration of the particles and other imperfections, if any.

3.3.1.2 COLD ROLLED SPECIMENS: At least sixteen pieces, eight each in longitudinal and transverse direction, are taken from each strip that is to be tested. These samples are then cold rolled to 10% and 20% reduction. The percentage deformation is kept at 20% maximum only. This is so because at higher reductions, strips begin to crack due to the inherent brittleness of the strips. Therefore, during cold rolling the desired deformation is obtained in successive passes. In each pass only 5% reduction is given.
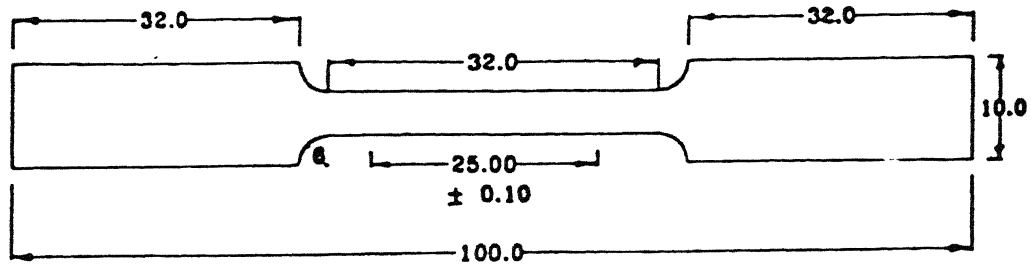
(a) ASTM standard tensile test specimen
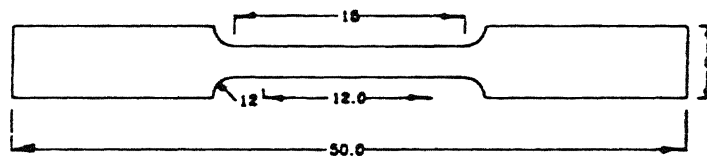
(b) British standard tensile test specimen [17]

All DIMENSIONS ARE IN mm

Fig. 3.3 : Tensile test specimens

Cold work in % deformation	Temperature for complete recrystallization (°C)
10	460
20	400
30	380
40	360
80	320
98	300

Table 3.3 : Recrystallization termpearature for aluminium [18]

3.3.1.3 ANNEALED SPECIMENS: As it is desired that the behaviour of the composite strips be tested in cold rolled as well as annealed conditions, some cold rolled samples are subjected to annealing. The recrystallization temperature is chosen depending on the percentage of deformation given to the samples. The deformation and corresponding recrysallization temperatures are given in Table 3.3. Annealing is carried out in a resistance furnace. The tensile testing specimens are then prepared from these heat treated samples.

3.3.1.4 HOT ROLLED SPECIMENS: At least four samples cut in the longitudinal and transverse directions from each strip, are heated to 400 °C in a resistance furnace, for about 15 minutes. These heated samples are then hot rolled on a two high rolling mill, rolling direction is kept parallel to the longitudinal direction of the strip. These two samples are subjected to 10% and 20 % deformation, respectively. The maximum deformation is limited to 20 % keeping in view the maximum deformation given during the cold rolling. This is done to have comparative data for both types of rolling. Here also the deformation is given in successive steps, with steps of 5% deformation. The time lag between the two successive passes is kept as low as possible to avoid the heat losses from the samples, thus ensuring that the rolling temperature is above the recrystallization temperature during the entire rolling process. The samples are allowed to cool down in the ambient conditions. These hot rolled strips are then taken for making tensile test specimens.

3.4 MECHANICAL PROPERTIES MEASUREMENT

3.4.1 Strength and Ductility

Tensile testing is carried out on the INSTRON 1195 tensile testing machine. In these tests, automated load verses displacement plots are recorded and the ultimate tensile strength, 0.2% yield strength and percentage elongation are calculated.

3.5 MICROSCOPIC EXAMINATION

3.5.1 Optical Microscopy

To observe the distribution of the reinforcing particles in the matrix first of all optical microscopic examination of the specimens is carried out. For this samples are cut from the cast strip with hack saw. Four to five samples from each strip are cut. Care is taken that these

samples are from different positions of the strips. It is done so as to know whether or not the distribution of the particles is uniform throughout the strip.

The samples are mounted using cold setting compound. The usual grinding and emery polishing is done with emery papers of grade 01 through 04. Then cloth polishing is done-first with 1 μ and finally with 0.3 μ alumina suspension. The cloth polishing is continued until a mirror surface is obtained. These samples are then employed for microscopic examination.

For the study of grain structure and the effect of rolling, the samples are taken from the broken specimens of the tensile testing. All the steps mentioned above are repeated in this case also Additionally, after cloth polishing, etching is carried out with Keller'e reagent

3.5.2 Scanning Electron Microscopy

The samples used for the SEM examination are the same as those used for the optical microscopy. The SEM examination is carried out on the JEOL JSM 840 machine. The main attention is focused on the distribution of the reinforcing particles, pores and the inclusions. The composition analysis is also carried out at various locations in the matrix. Photographs are taken at various magnifications to reveal the nature of the distribution of the ceramic particles and also to see the effect of the rolling operations.

The tests carried out and the examinations made on the strips are discussed in detail in the next chapter on results and discussions.

Chapter 4

RESULTS AND DISCUSSION

Using experimental procedure described in the previous chapter, several experiments were carried out to examine the distribution of the reinforced particles in the cast strips and also the effect of operational parameters, e.g. speed of rotation of the caster drum, superheat, etc. on the properties of the strip. The behaviour of the strips under as-cast, cold rolled, annealed and hot rolled conditions is investigated The fractured surface after testing is also observed. The results obtained are discussed in this chapter.

4.1 DISTRIBUTION OF PARTICLES

The distribution of reinforcing particles in the metal matrix being the single most important factor, from the application point of view, it is examined using optical and scanning electron microscope. Figure 4.1 shows the particle distribution. It is seen that the reinforcing particles are fairly evenly distributed in the aluminium matrix. Around 80% of the particles added (5 wt% of the matrix) were embedded in the matrix while remaining 20% floated onto the melt surface.

4.2 AS-CAST STRIPS

The strips produced are observed visually and then under the optical microscope. These strips are then tested for mechanical properties in the as-cast condition. The results obtained are discussed in this section.

20 RPM, 60 °K superheat

26 RPM, 60 °K superheat

Fig. 4.1 : Micrographs showing distribution of particles in the matrix

4.2.1 Surface Quality

From the visual examination it is observed that the roll side surface is smooth while the top side surface is comparatively rough. This is in agreement with the study carried out by Fao b.₹. on aluminium strips [19] The degree of roughness is more dominant at lower drum speeds as compared to that at higher speeds. Transverse cracks are also dominant at lower drum speeds The surface is relatively smoother if the strip is cast from a melt having higher superheat From the microstructural point of view, lower degree of superheat is preferable because it produces equi-axed structure But when the melt superheat is very low, there is the danger of choking of the tundish nozzle which disrupts the whole process. The reason for increase in the surface roughness at lower speeds of rotation and lower melt superheat may be attributed to large strip thickness in both the cases Thicker strip section offers more resistance to the heat transfer resulting in reduced solidification rate. This adversely affects the surface quality. Strips of larger thickness are also more prone to transverse cracking due to larger bearing stresses that are developed due to the straightening and peeling off the solidified strip from the drum. Also the stirring of the liquid metal during the incorporation of the reinforcing particles contributes to the roughness of the strip. The stirring entraps the atmospheric gases in the melt which may try to escape during the solidification process leading to rough strip surface

4.2.2 Tensile Strength And Ductility

The tensile strength in the longitudinal direction (both, ultimate strength as well as 2% yield strength) is tested for the strips produced at various caster drum speeds and with different degrees of superheats. The effect of these parameters on the strength and ductility is discussed in this section

4.2.2.1 EFFECT OF SPEED OF ROTATION OF THE CASTER DRUM: Speed of the rotation of the caster drum is the one of the most influencing factors regarding the properties of the strip. It also controls the rate of production of the strips. Figure 4.2 shows the effect of speed of rotation of caster drum on the tensile strength of the strip with a superheat of 60 °C and water flow rate of 0.4 GPM. Figure 4.3 shows variation of
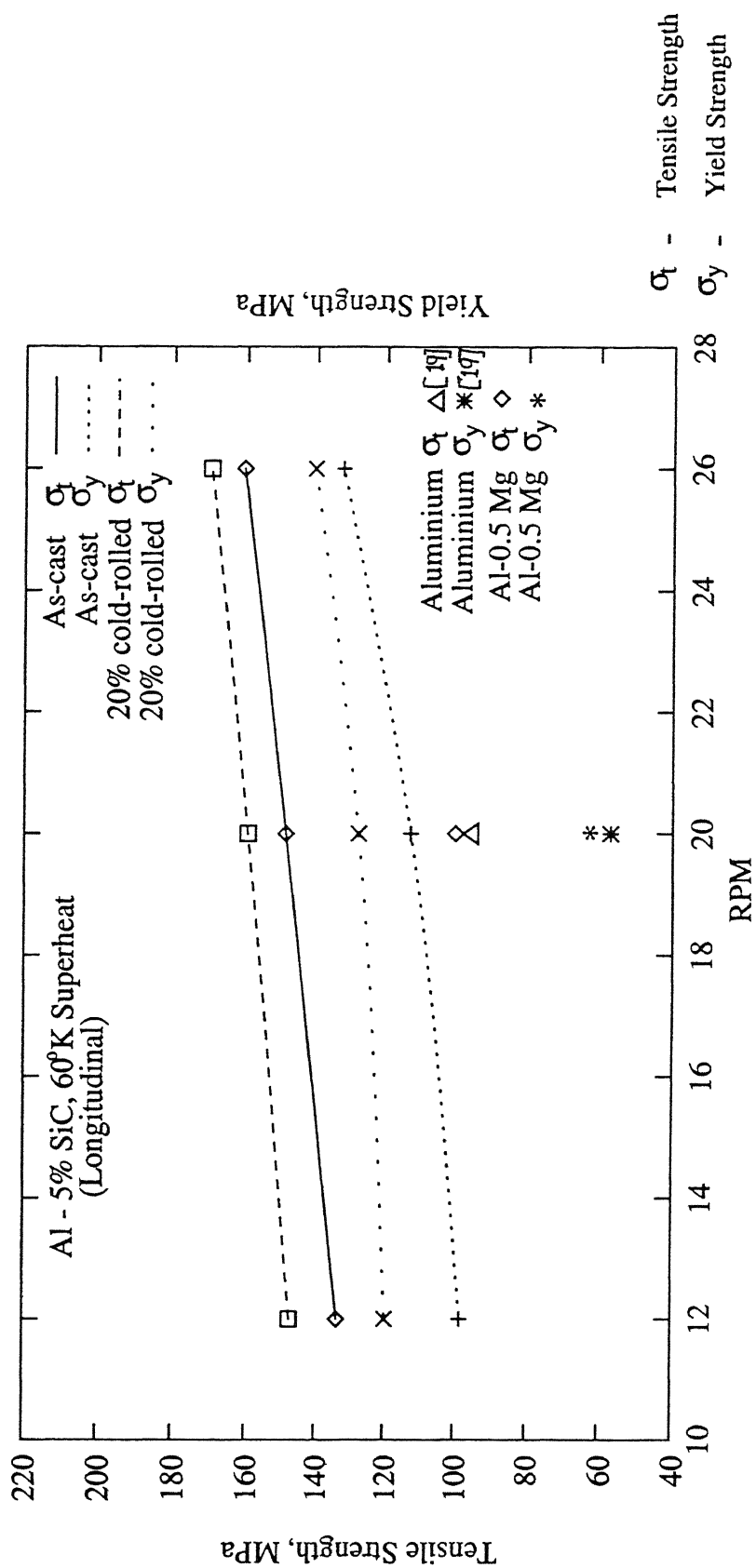
Fig. 4.2 : Effect of RPM on strength of as-cast and cold-rolled strips in longitudinal direction
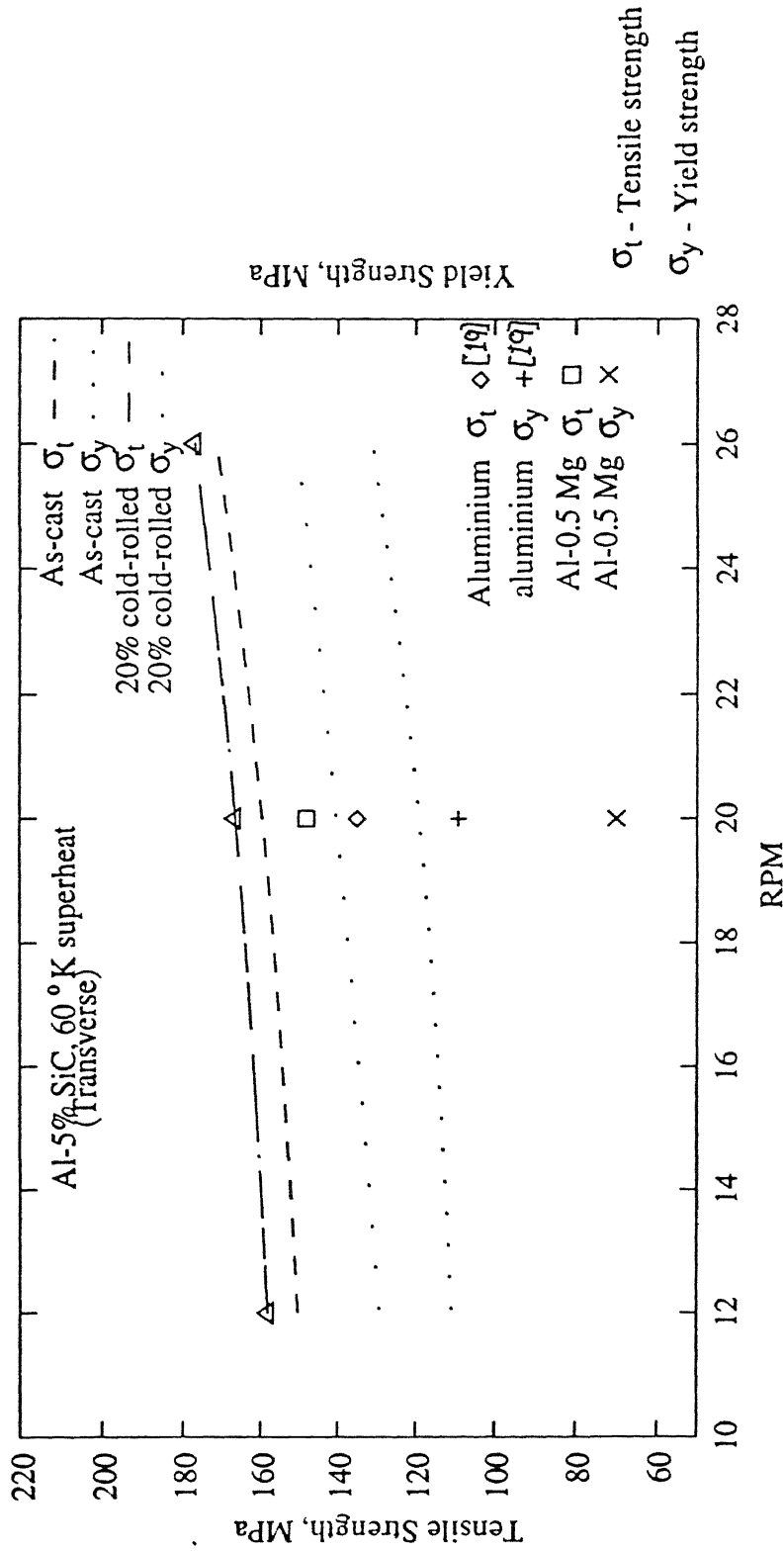
Fig. 4.3 : Effect of RPM on strength of as-cast and cold rolled strips in transverse direction

transverse strength as a function of speed of rotation Both these behaviours follow a similar trend as that has been reported by ~~~ ~,~ for aluminium [·]. It is seen that the ultimate tensile strength increases from 144.6 MPa to 164.68 MPa when the speed of rotation is changed from 12RPM to 26 RPM. The reason for increase in strength at higher speeds can be attributed to the increased rate of solidification which, in turn, affects the grain size. The thermal gradient between the drum surface and the liquid metal establishes the requisite heat flow during solidification. This gradient is a function of melt temperature (i. e superheat), the drum surface temperature and the heat transfer coefficient across the metal/ drum interface. For a rotating drum the contact area increases linearly with increase in the speed of rotation of the drum This affects the rate of heat withdrawal from the molten metal pool. It should be noted that the rotation of the caster drum has two effects: it increases the heat transfer coefficient at the metal/ drum interface because of the enhanced fluid flow in the metal pool, and new surface is formed continuously between the caster drum and the solidifying strip.

Both, the increased mechanical contact between the drum and the strip and the enhanced heat transfer coefficient because of the higher rate of heat withdrawal, produce a product with fine grain structure at higher rotational drum speeds. The effects of the speed of rotation on the ductility of the strips in longitudinal and transverse directions are shown in Figs 4 4 and 4.5. The ductility in longitudinal direction decreased from 2.26 % to 1.6 % for variation in the speed of rotation of the drum from 12RPM to 26 RPM. For transverse direction similar trends are observed The reduction in the ductility of the strips at higher speed of rotations is partly due to the increased porosity of the strips and partly due to the smaller grain size factor.

4.2.2.2 EFFECT OF MELT SUPERHEAT: Figs 4.6 and 4.7 show the effect of melt superheat on the tensile strength and ductility of the strips in the as cast condition. 60 °C, 90 °C and 120 °C are three superheats selected for the study. Graphs of tensile strength and percentage elongation at these three superheats at constant drum speed of 20 RPM and water flow rate of 0.6 GPM are shown in the above figures. These confirm with the results for aluminum strips[19]. It is seen that the ultimate tensile strength decreases from 148.7 MPa to 119.9 MPa with increase in the superheat from 60 °C to 120 °C. The ductility also decreases from 1.8% to 1.6%. The reduction in the strength is due to an
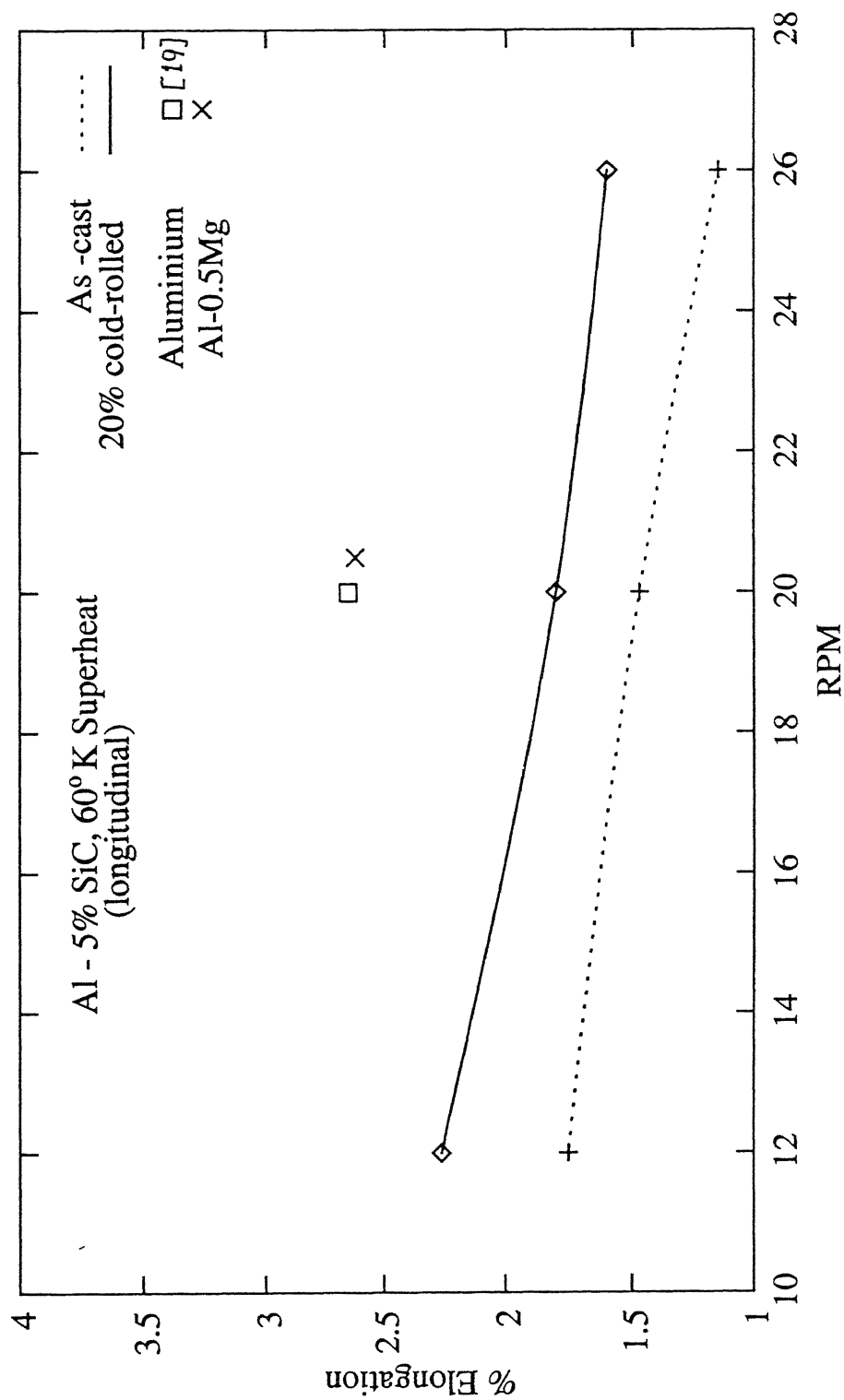
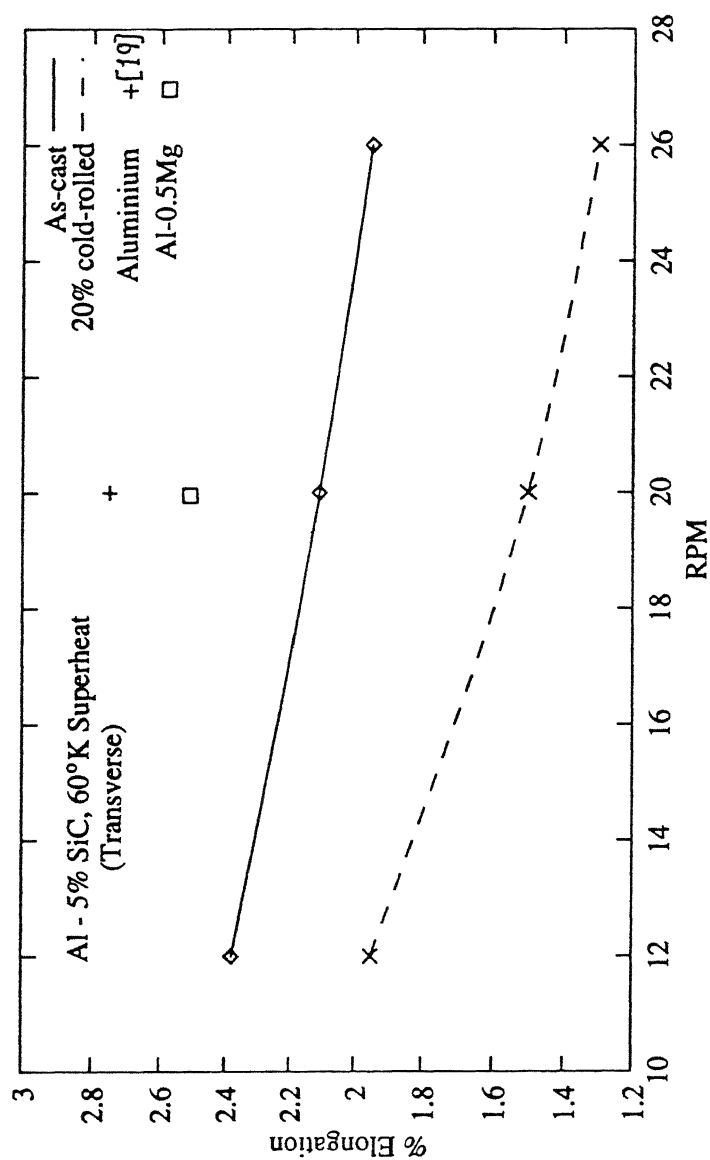**Fig. 4.4 : Effect of RPM on ductility of as-cast and cold-rolled strips in longitudinal direction**

Fig. 4.5 : Effect of RPM on ductility of as-cast and cold rolled strips in transverse direction
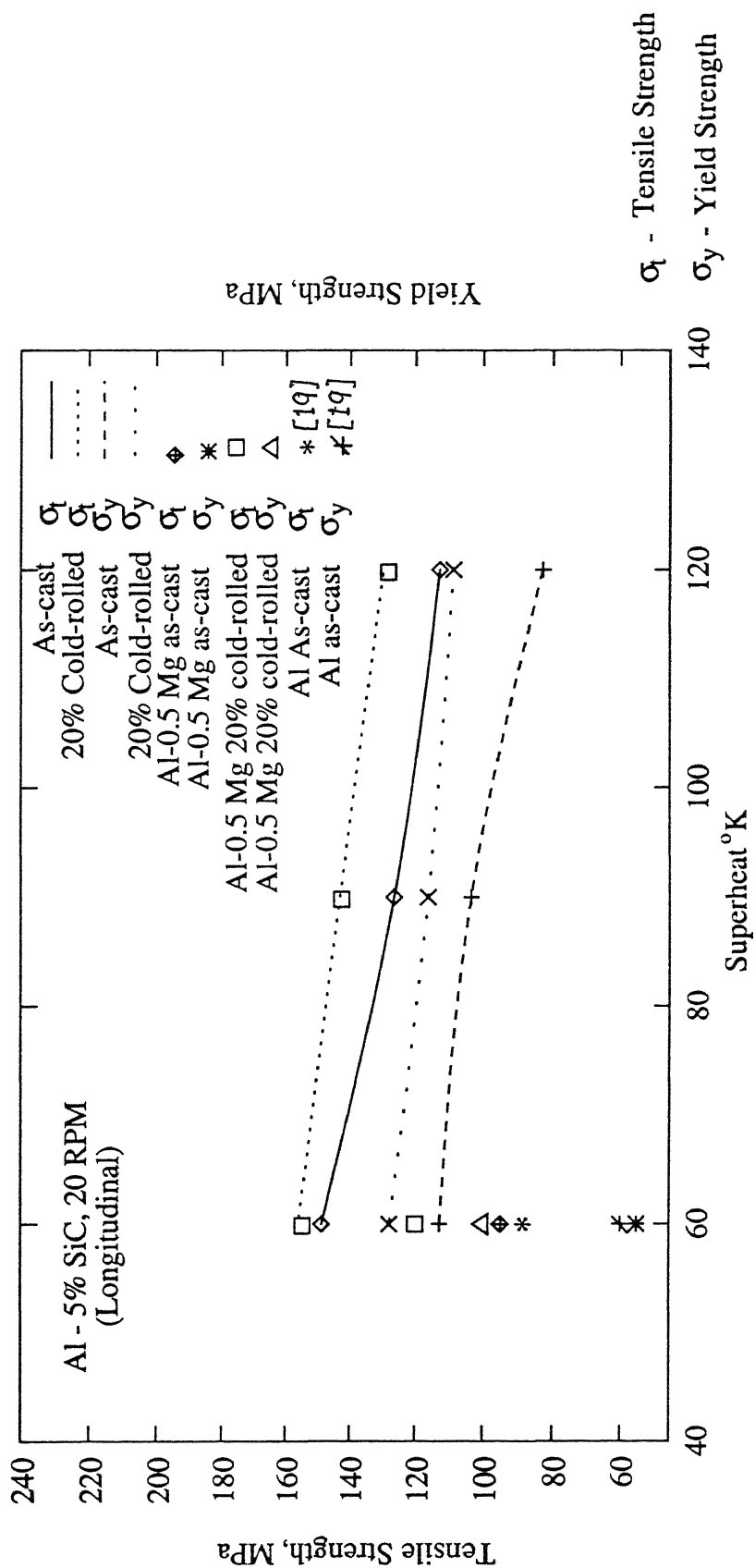
Fig. 4.6 : Effect of superheat on strength of as-cast and cold-rolled strips in longitudinal direction
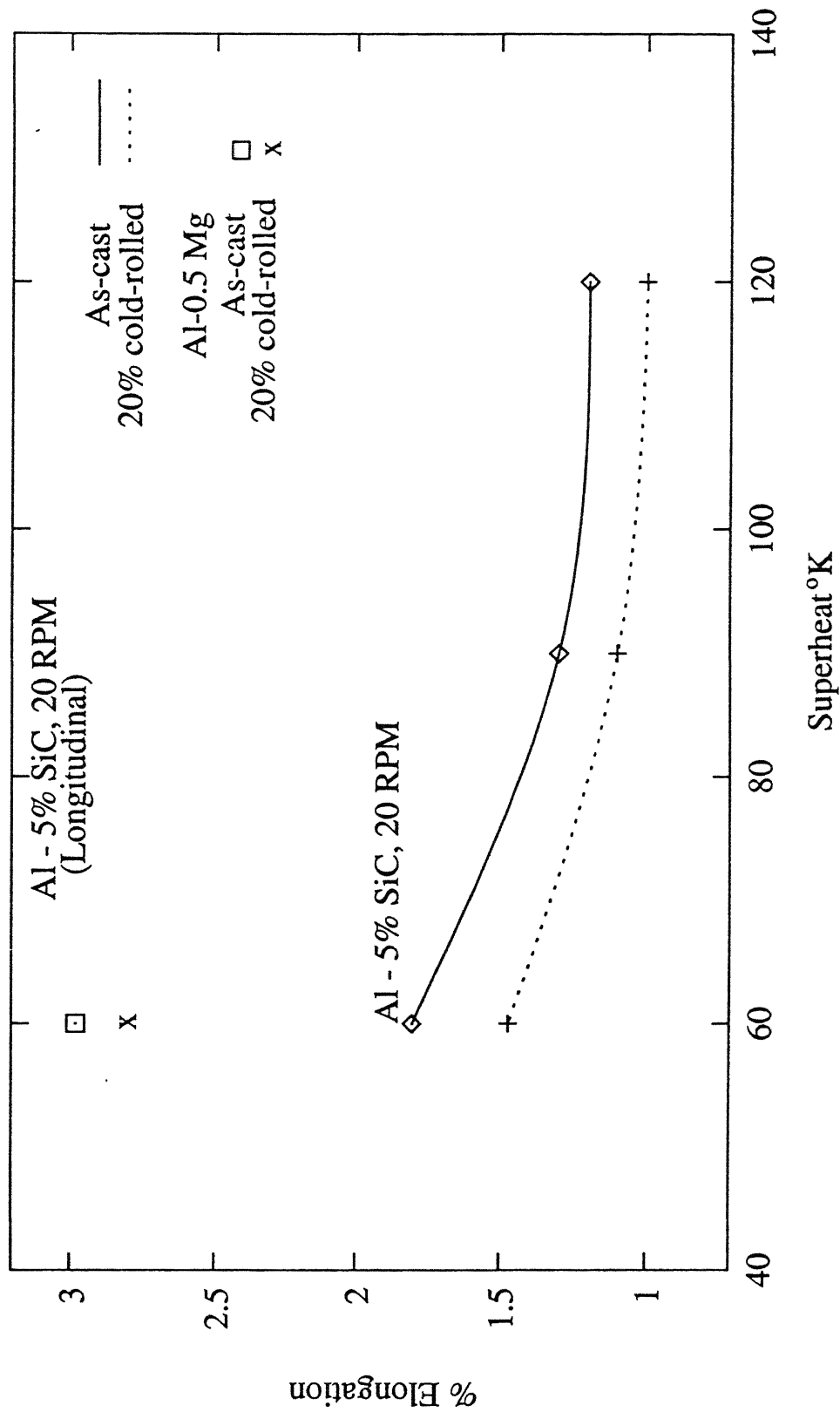
Fig. 4.7 : Effect of superheat on ductility of as-cast and cold-rolled strips in longitudinal direction

increase in the grain s. .e at higher melt superheats. The higher porosity resulting from higher degrees of superheats is likely a reason for lesser ductility at higher superheats.

Figure 4.8 shows the micrograph of the fractured specimen after tensile testing. It shows that the fracture has taken place along the interface between the matrix and SiC particle. It is due to the reduced cohesive strength at the particle/matrix interface. Improper wetting can be attributed as a reason for this reduced strength.

4.2.3 Microstructure And Internal Quality

The porosity is one of the important factors determining strength and ductility of the strips produced. It depends on the following factors-

1 solidification rate
2. speed of rotation of drum
3 degree of superheat
4 stirring

4.2.3.1 EFFECT OF SOLIDIFICATION RATE: As is known, volume of a liquid decreases on solidification. For aluminium melt, this shrinkage on solidification is 6% to 8 %. For the commercial aluminium used in the experiments this might be still higher. If the solidification rate is high enough not allowing the dissolved gases to escape, because of this shrinkage the cavities are produced which increase the porosity of the final product.

4.2.3.2 EFFECT OF SPEED OF ROTATION OF DRUM: The speed of the caster drum significantly affects the internal porosity. It can be seen from the SEM photograph in Fig. 4.9 that the strip produced at 20RPM is less porous as compared to the strip produced at 26 RPM This can be due to higher rate of solidification at higher speeds that allows lesser time for the entrapped gases to come out of the melt.

4.2.3.3 EFFECT OF MELT SUPERHEAT: Variation in porosity of the strips with superheat is shown in Fig. 4.10. As can be seen, the porosity is more for the strip which is cast from melt having higher degrees of superheat. This is due to two reasons:
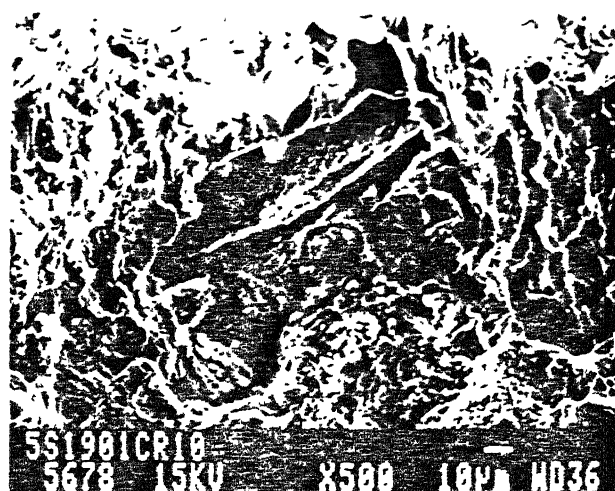
20 RPM, 60 °K superheat

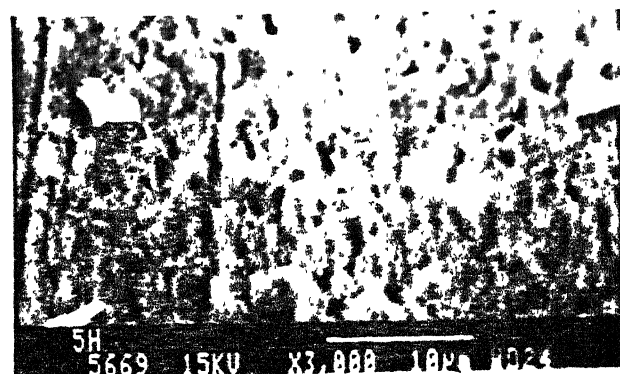**Fig. 4.8 : Micrograph of fractured surface of as-cast strip**

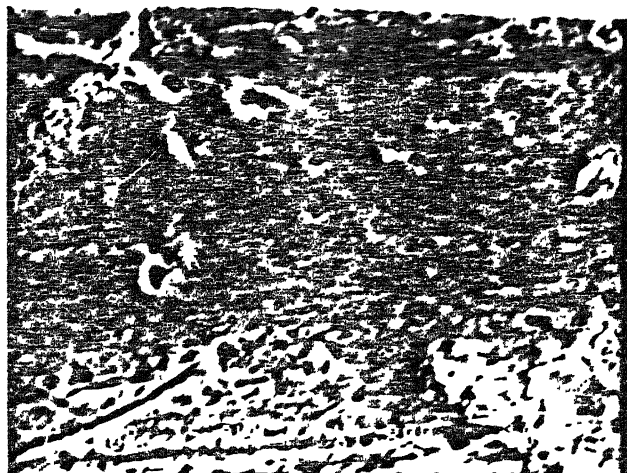

20 RPM, 60 °K superheat

26 RPM, 60 °K superheat

Fig. 4.9 : Effect of RPM of the caster drum on internal porosity
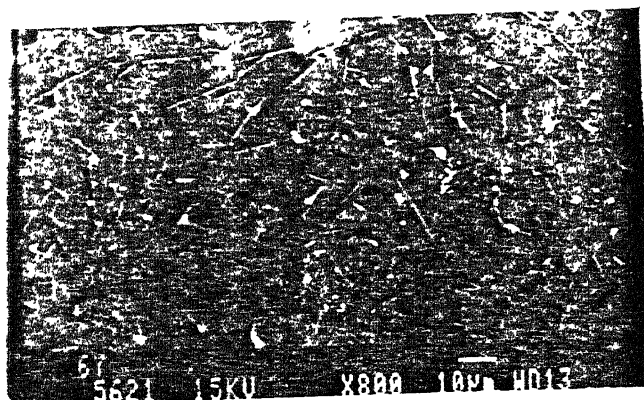
12 RPM, 60 °K superheat

12 RPM, 120 °K superheat

Fig. 4.10 : Effect of melt superheat on internal porosity

20 RPM, 90 °K superheat

**Fig. 4.11 : Micrograph showing segregations in matrix in as-cast strip**

(i)   the solubility of the gases in the molten metal is higher at higher temperature, and

(ii)   the degree of volume shrinkage also increases with increase in the melt superheat.

4.2.3.4 EFFECT OF STIRRING: One of the sources of porosity in the strips is stirring of the melt. As the melt is stirred in the open air, atmospheric gases get entrapped in it. Due to the vigorous stirring these gases do not get chance to escape from the melt even on cooling, thereby increasing the porosity. This can be reduced by employing lower stirrer speeds. But this will be at the cost of reduced wettability between the particles and the metal.

## 4.2.4 Segregations in the Matrix

The SEM photograph shown in Fig. 4.11 reveals that there is segregation of some elements like Fe, Cu, Si, Mn, etc. in different quantities as is seen in the figure in rod like forms. The compositional analysis shows that the amount of iron is about 0.8% and the other elements account for other 1 to 1 5%. As aluminium used is of commercial purity, some percentage of these impurities is there in the metal right from the start. High amount of iron may also be due to the iron rod used to remove the dross from the melt. Further, some amount of iron is also added to the melt from the stainless steel shaft of the stirrer. Iron forms second-phase particles such as Al_3Fe in presence of aluminium. Silicon also forms α-AlFeSi and β-AlFeSi phases [21]. These second-phase constituents segregate between the dendrite arms during solidification In Fig. 4.11, these phases are seen as bright rod like structures as well as bright spots in the matrix. The second phase pins down the grain boundaries thereby preventing fully formed grain structure. During solidification these phases grow over the grains of alumunium thus covering it partially as is clear from the photograph.

The micrographs also show the presence of some non-metallic inclusions. The major source for these inclusions is the erosion of the refractory material of the crucible used to prepare the composite melt. This is due to the prolonged stirring (15-20 minutes) of the composite melt during and after the incorporation of the reinforcing particles in molten aluminium. Although care was taken to minimize the erosion of crucible walls, some erosion was inevitable. The other source of these inclusions is the other non-metallic phases. These inclusions adversely affect the mechanical properties of the
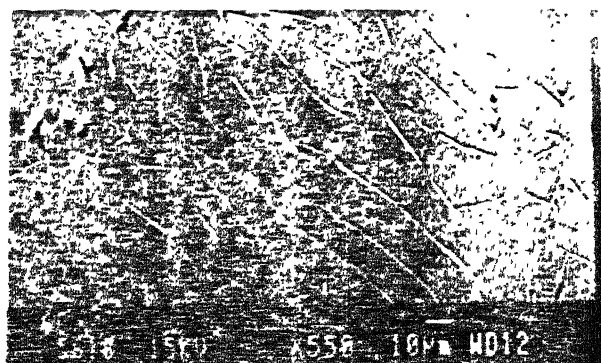
20 RPM, 60 °K superheat

Fig. 4.12 : Micrograph of as-cast strip in longitudinal direction
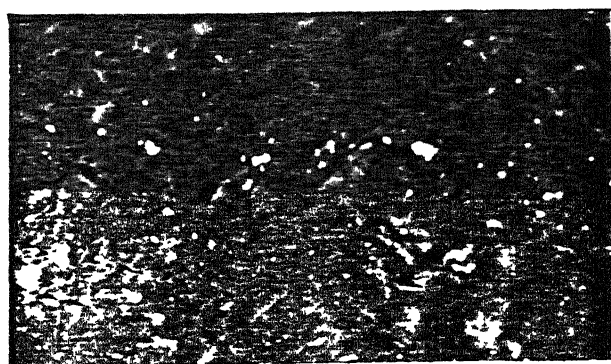
20 RPM, 60 °K superheat

Fig. 4.13 : Micrograph of as-cast strip in transverse direction

product During further processing (e.g rolling. forging, etc), the material may rupture due the inclusion nucleated fracture. As these inclusions can not be removed during the casting process, it is of great importance that the melt used for the casting is clean and adequately deoxidized

Figures 4.12 and 4.13 show SEM micrographs for the samples taken from the longitudinal and transverse sections respectively. The segregating phases are seen as rod like in Fig. 4.12 while spots are seen in the Fig. 4.13. This is due to the higher rate of heat removal in the direction in which the strip is cast. In the transverse section, this rate is comparatively lower across the thickness. This results in dominance of grain growth over the growth of the segregates in transverse direction. Although some rod like segregates are seen in the transverse section also.

4.3 COLD ROLLED, COLD ROLLED AND ANNEALED, AND HOT ROLLED STRIPS

From the point of view of studying the behaviour of the formed strips, the as-cast strips have been subjected to the following treatment·

◆ cold rolling

◆ cold rolling and annealing

◆ hot rolling

4.3.1 Cold Rolled Strips

4.3.1.1 STRENGTH AND DUCTILITY : The strength and ductility values obtained in the cold rolled condition show similar trends as for the cast specimens, albeit with some variations. As can be seen from Fig. 4.2, the strength increases from 154.2 MPa at 12 RPM to 168.4 MPa at 26 RPM. The ductility decreases with the increase in the speed of rotation of the caster drum as shown in Fig. 4.4. The increase in strength may be due to the work hardening effect during the cold deformation. The reduction in ductility is attributed to the increase in defects developed during the cold deformation. The strength and ductility behaviour with the superheat is also shown in Figs 4.6 and 4.7. It is seen that both, strength and ductility decrease with increase in the superheat.

20 RPM, 60 °K superheat

Fig. 4.14 : Micrograph of cold rolled strip

4.3.2.2 MICROSTRUCTURE AND INTERNAL QUALITY: Figure 4 14 shows the effect of cold rolling on the microstructure for the strips produced at 20 RPM and 60 °C superheat. These micrographs show the effect of segregation, inclusions and grain size. As can be seen, the segregation which are rod like in as cast specimens, are broken and finely distributed throughout the matrix. It can also be noticed that some inclusions of spherical shapes are present in the matrix As for the grain boundaries, these are not seen clearly. This may be due to the segregating effect of the metallic impurities in unusually higher amounts. These inclusions pin down the grain boundaries thereby avoiding the alignment of the grain boundaries.

4.3.2 Cold Rolled and Annealed Strips

The temperatures employed for the annealing for a particular degree of cold rolling are shown in Table 3.3. The recrystallization temperatures are taken for aluminium, ignoring the effect of reinforced particles and magnesium, which is present in small quantity.

4.3.2.1 STRENGTH AND DUCTILITY: The results obtained for this case are shown in Figs 4.15 and 4.16. It is seen that the strength is lesser than that of the as cast strips but the elongation is more. The grain refinement taking place due to annealing can be sited as one of the reasons for both these bahaviours. It is found that the strength increases from 122.8 MPa for 10% cold rolled and annealed to 140.0 MPa for 20% cold and annealed. The percentage elongation decreases from 4 12 at 10 % deformation to 2 75 at 20% deformation. It is found that both the strength and ductility decrease with increasing superheat. This behaviour is in confirmity with that of aluminium strips [19]. The behaviour of strips in annealed condition with superheat is shown in Fig. 4 17. The tensile strength decreases with the superheat.

Figure 4.18   shows the fractured surface for the strip produced at 20 RPM and 60 °C superheat under the annealed condition. It shows that the fracture occurred in the ductile manner.

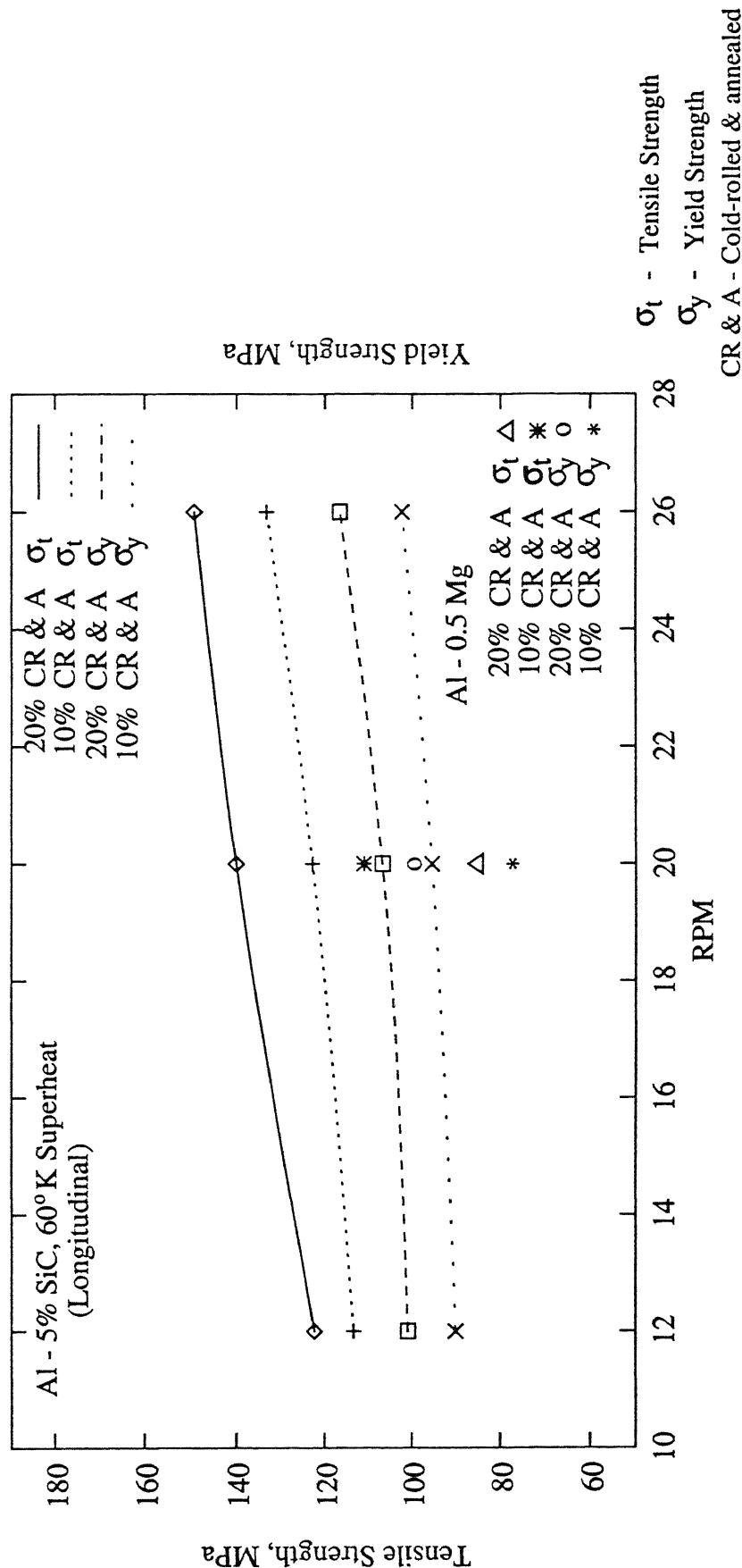**Fig. 4.15 : Effect of RPM on strength of annealed strips in longitudinal direction**
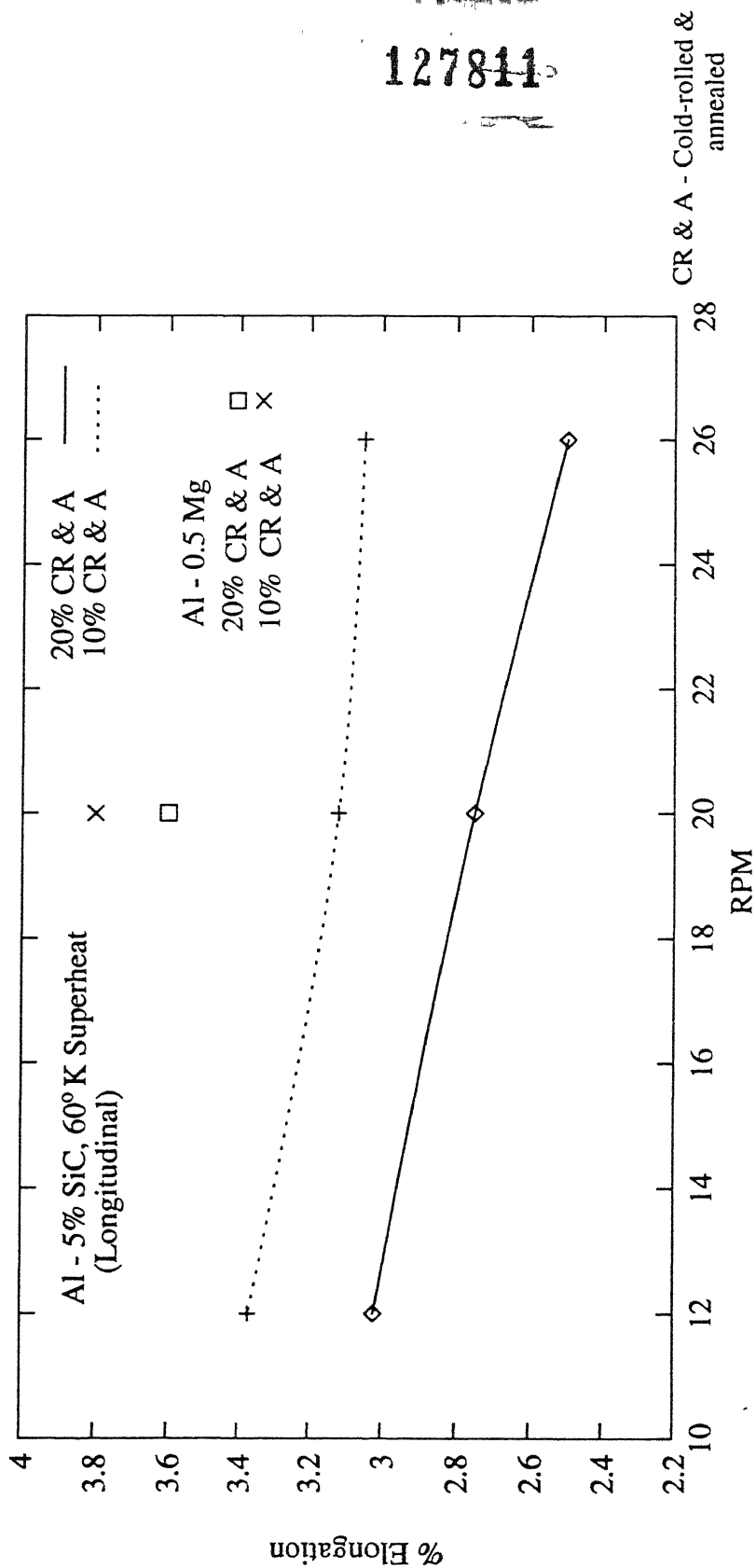
Fig. 4.16 : Effect of RPM on ductility of annealed strips in longitudinal direction

CR & A - Cold-rolled & annealed

Fig. 4. 17 : Effect of superheat on strength of annealed and hot-rolled strips in longitudinal direction
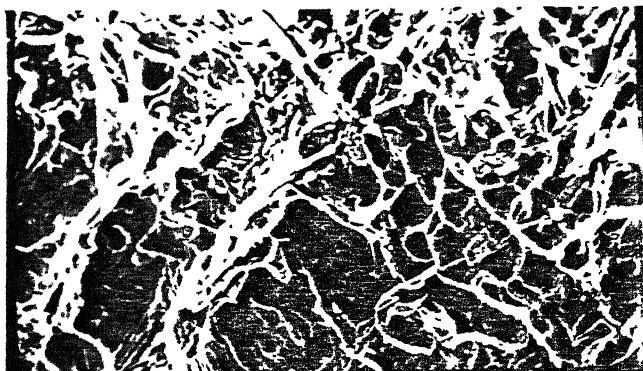
26 RPM, 60 °K superheat

Fig. 4.18 : Micrograph of fractured surface of cold rolled and annealed strip

4.3.2.2 MICROSTRUCTURE AND INTERNAL QUALITY: In this case it is seen that the segregates which are long and elongated in the as-cast material are broken and finely distributed throughout the matrix. The grains can not be seen clearly because of the effect of the metallic impurities like iron

4.3.3 Hot Rolled strips

4.3.3.1 STRENGTH AND DUCTILITY: Figures 4.19 and 4.20 show the longitudinal and transverse strengths as a function of the drum speed. It is seen that the tensile strength increases with RPM for a given percentage of deformation. This is similar to the behaviour of the hot rolled aluminium strips [20] Also from Fig. 4.19 it is evident that the tensile strength is more for strips with 20% deformation than those for 10% deformation for a given rotational speed of the drum. The strength in transverse direction is higher than that in the longitudinal direction. Comparing the results of cold rolled and annealed and only cold rolled strips, it is seen that the strength for the strip produced at 20 RPM and 60 °C superheat with 20% deformation is more than that for the strip cold rolled and annealed at the same working conditions, viz. 20 RPM and 60 °C superheat with 20% deformation The higher strength in hot rolled specimens can be attributed to:

- Formation of smaller grains at higher deformation
- Annihilation of pores

Figure 4.17 shows tensile strength in longitudinal direction as a function of melt superheat at 20 RPM of drum speed The strength decreases with the superheat. This may be due to the higher amount of porosity at higher degree of superheat which leads to the internal cracking.

Figure 4.21 shows ductility in the longitudinal direction as a function of drum speed. It can be seen that ductility decrease with the drum RPM. It also shows that the ductility decreases with increasing percentage deformation. Figure 4.22 shows the effect of superheat on ductility at 20 RPM. The ductility decreases with increase in the superheat. This may be, as mentioned earlier, due to higher porosity at higher superheats.
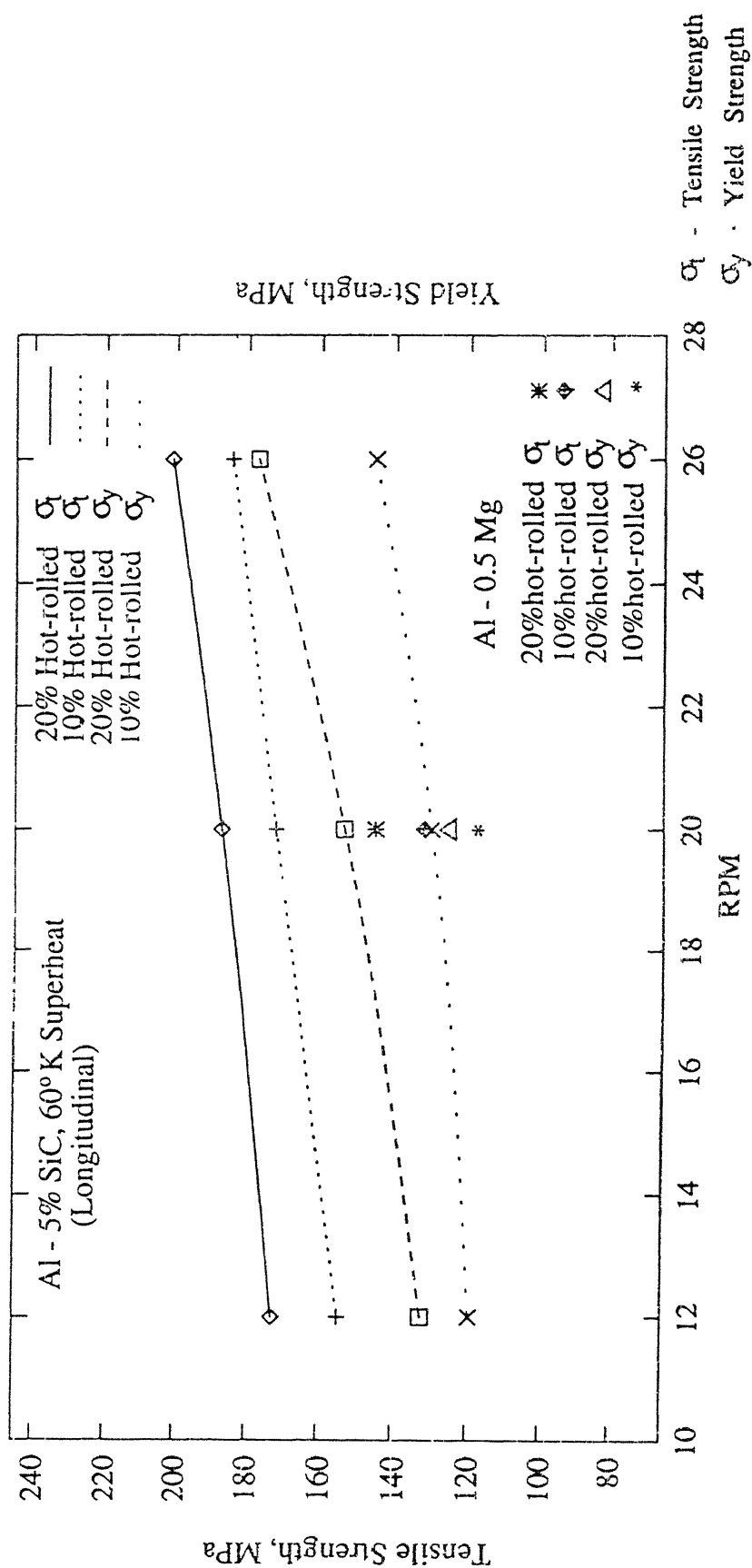
Fig. 4.1.9 : Effect of RPM on strength of hot-rolled strips in longitudinal direction
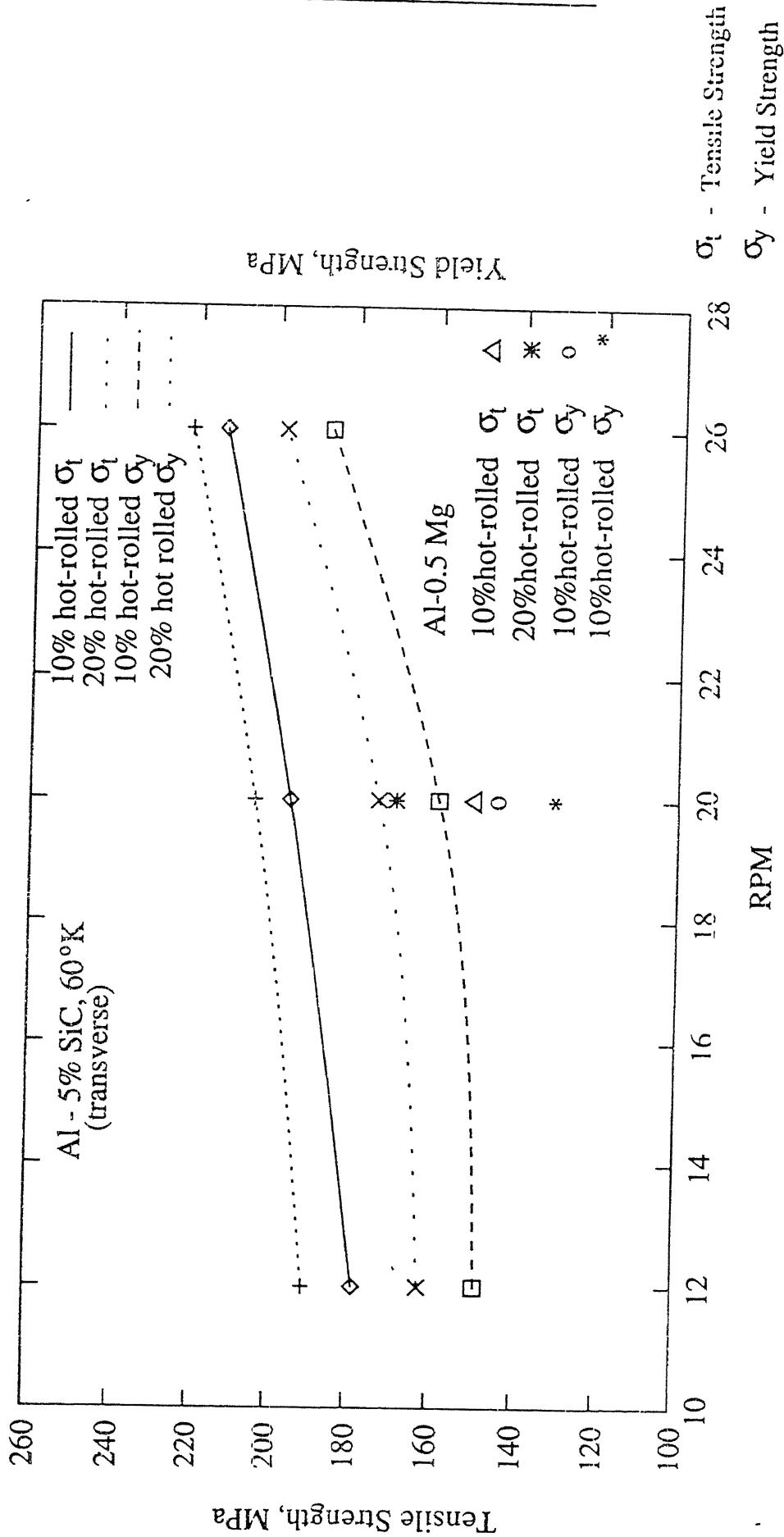
Fig. 4. 20 : Effect of RPM on strength of hot-rolled strips in transverse direction
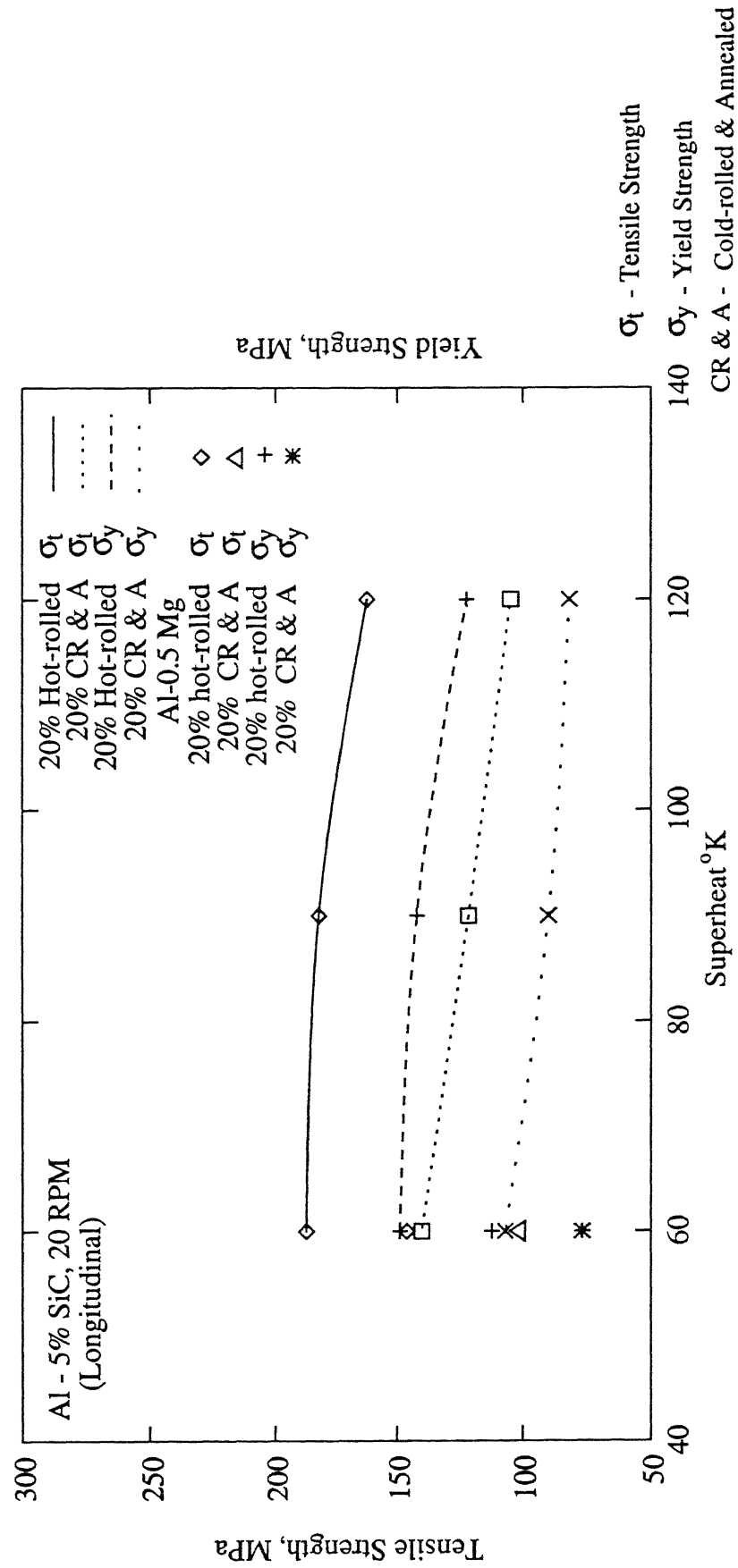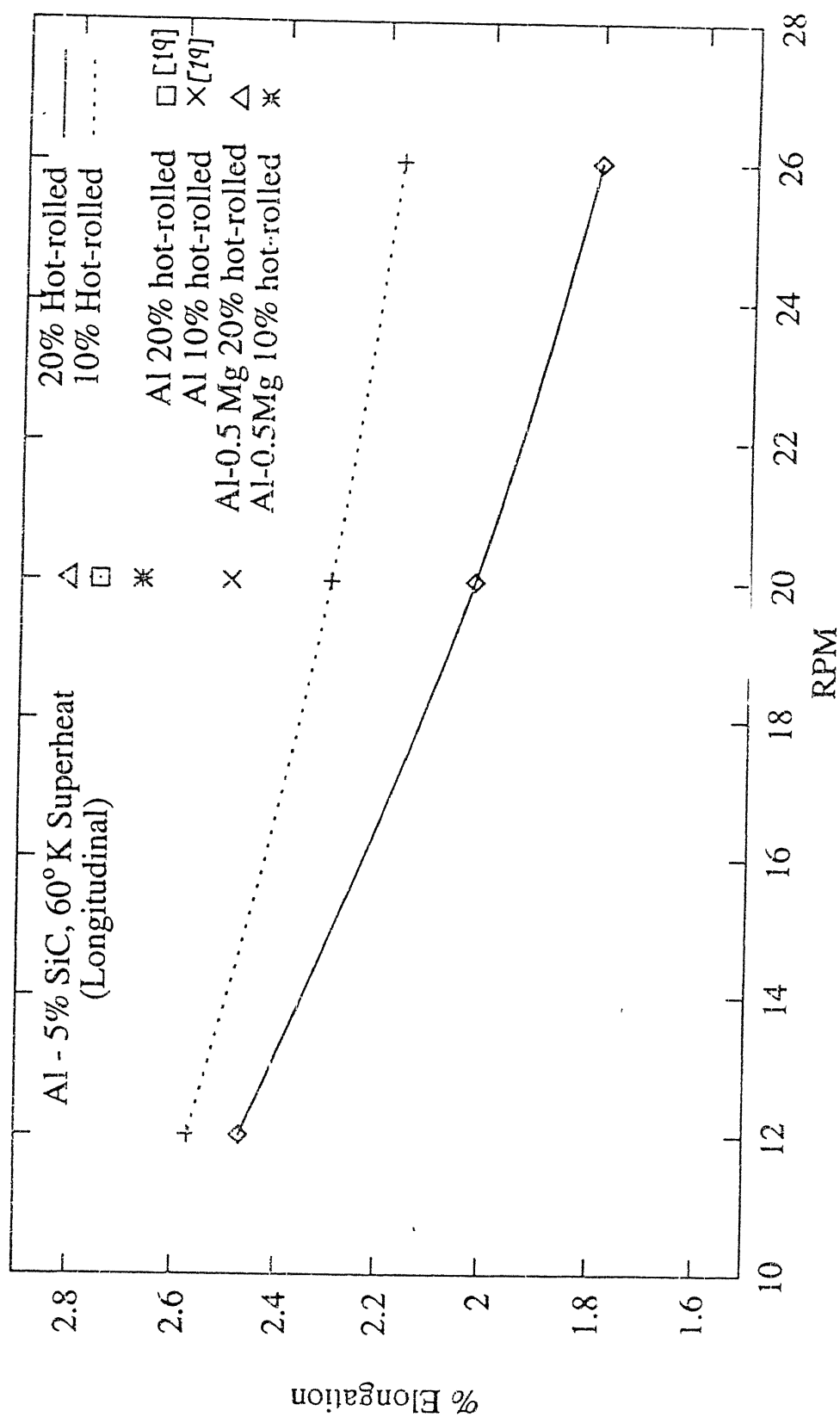
Fig. 4.21 : Effect of RPM on ductility of hot-rolled strips in longitudinal direction
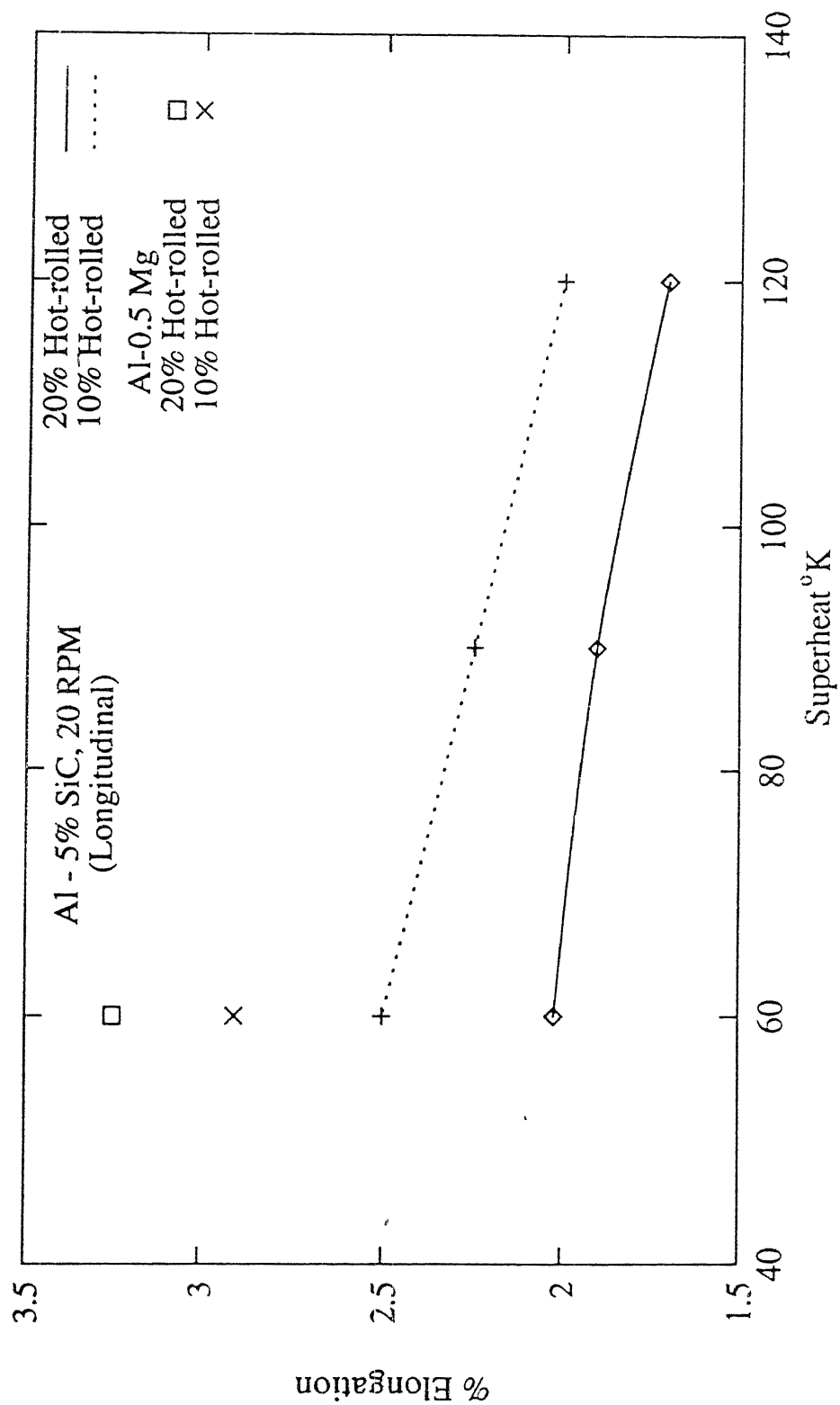
Fig. 4.22 : Effect of superheat on ductility of the hot-rolled strips in longitudinal direction
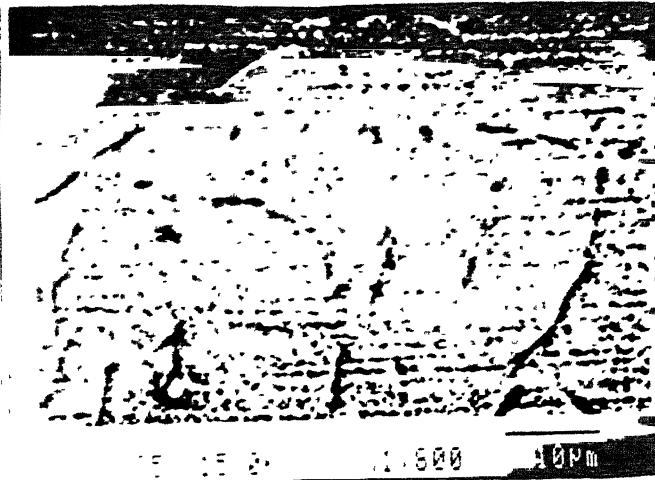
20 RPM, 60 °K superheat   20 RPM, 90 °K superheat

Fig. 4.23 : Micrographs of hot rolled strips
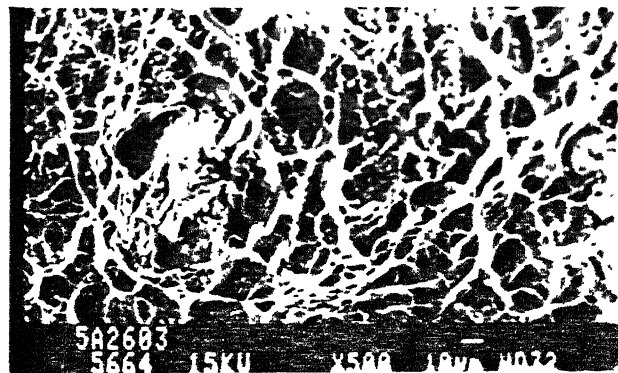
26 RPM, 60 °K superheat

Fig. 4.24 : Micrograph of fractured surface of hot rolled strip

Instead of annihilation of the pores on hot rolling, internal cracking of the strips is observed giving lower elongation. The extent of internal cracking increases with increased deformation giving lower ductility.

4.5.2 Microstructure and Internal Quality

It is observed that the segregates in this case also, as in cold rolled specimens, are broken and finely distributed. The grains are not clearly defined because of the pinning effect of the impurities present in the matrix material. Internal cracks are observed for 20 RPM and 90 °C superheat. Figure 4.23 shows the microstructure for hot rolled strips at 20 RPM and 60 °C and 90 °C respectively. It can be clearly seen that the porosity is higher in the later case. Also partial recrystallization of aluminium is observed. This is due to the less available time for complete recrystallization during the hot rolling process.

Figure 4.24 shows micrograph of the fracture surface of hot rolled sample. It shows that the fracture occurred in a ductile manner.

Chapter 5

SUMMARY AND CONCLUSIONS

The present investigation primarily involves two stages. In the first stage, composite strips of aluminium reinforced with SiC particles have been produced using single roll continuous strip caster (SRCSC)-designed and developed in our laboratory by Mehrotra and coworkers essentially for producing near-net-shape metal strips. Alloying of aluminium matrix with magnesium, preheating of the particles, and stirring of the melt are employed for improving the wetting characteristics of the particles with aluminium melt Vortex method has been used for preparing the composite melt In the second stage, evaluation of microstructure and mechanical properties has been carried out. Microstructural investigations are based on optical and scanning electron microscopy to study the distribution of particles in the cast strips and internal quality of the strips. Mechanical properties such as strength and ductility are examined for the composite strips produced at various operating conditions such as the speed of rotation of the caster drum, melt superheat, etc Effect of these parameters on the surface quality of the composite strips have also been examined The behaviour of the composite strips in as-cast, cold rolled, cold rolled and annealed, and hot rolled conditions in relation with the operating parameters is examined.

More than 50 experiments have been performed during the course of this investigation. From the analysis of the experimental data, the following conclusions are drawn:

i. It is possible to incorporate SiC particles in aluminium melt with proper preheat treatment of the particles and with addition of surface active alloying elements such as magnesium in the metal matrix.

ii. The composite melt causes severe erosion and corrosion of the stirrer blades of the uncoated stainless steel stirrer. Stirring with a graphite stirrer is, therefore, desirable

iii. It is not possible to incorporate SiC particles of sizes less than 100 μ into the aluminium melt.

iv. The roll side surface of the composite strip is smooth as compared to the top side surface. The degree of roughness increases with increase in the melt superheat.

v. Brittleness of the strips increases when higher amount of magnesium is added to the aluminium melt.

vi. Occurrence of transverse cracks is more in case of thicker strips.

vii. Considerable amount of segregations of iron and other metallic impurities are present in the strips. Non-metallic inclusions are also present. Grain structure is not developed properly due to the presence of these segregations.

viii Internal porosity in the strip increases with increasing rotational speed of the caster drum, as well as with increasing melt superheat.

ix Strength of the composite strips is about 25% more than that of aluminium strips produced using the same technology.

x The strength of the composite strips increases while ductility decreases with increase in the speed of rotation of the caster drum.

xi The strips are prone to cracking at deformations more than 20% during cold rolling

xii. With an increase in the degree of deformation there is an increase in the strength of cold rolled and hot rolled strips. Ductility, however, decreases with increased degree of deformation.

xiii. Strips produced from melts with higher superheats are more prone to cracking, during hot rolling of the strips.

Appendix A

SINGLE ROLL CONTINUOUS STRIP CASTER

The design and fabrication of single roll continuous strip caster was initiated by Mehrotra and co-workers [21-24]. The fabrication of the caster was completed, and it was made fully operational in producing aluminum strips at various operating conditions. The same caster was used for producing MMC strips in this investigation. A brief description of caster design and production method of strips is presented here

A schematic representation of the caster assembly is shown in Fig. A.1. The main components of the caster include:

  i.   tundish/ reservoir

  ii. caster drum assembly

  iii. water spray system

  iv. knife-edge

A.1 TUNDISH/RESERVOIR

Tundish, which is made up of a fire clay brick, primarily acts as a reservoir to hold the molten metal, and feeds it to on to the rotating drum in a controlled manner. The rate of flow of the metal to the drum, which is determined by the rate at which the metal is removed in the form of the solidifying strip, is controlled by controlling a constant metal head in the tundish. The tundish wall facing the drum has the same concentric profile as that of the drum. It has a rectangular opening at the bottom of this curved face that acts as an outlet for the molten metal
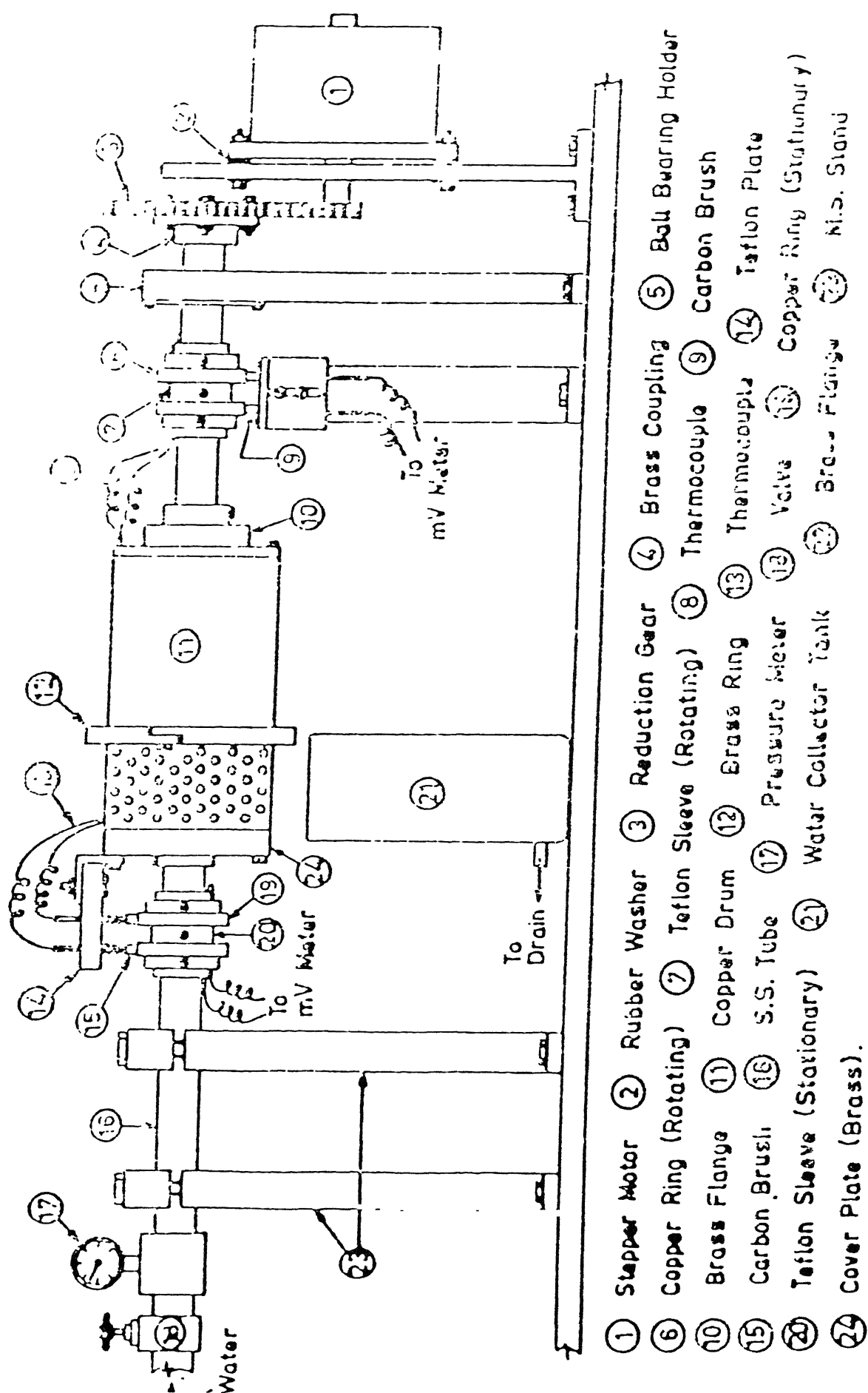
Fig. A.1 : Schematic diagram of Single Roll Continuous Strip Caster

① Stepper Motor ② Rubber Washer ③ Reduction Gear ④ Brass Coupling ⑤ Ball Bearing Holder

⑥ Copper Ring (Rotating) ⑦ Teflon Sleeve (Rotating) ⑧ Thermocouple ⑨ Carbon Brush

⑩ Brass Flange ⑪ Copper Drum ⑫ Brass Ring ⑬ Thermocouple ⑭ Teflon Plate

⑮ Carbon Brush ⑯ S.S. Tube ⑰ Pressure Meter ⑱ Valve ⑲ Copper Ring (Stationary)

⑳ Teflon Sleeve (Stationary) ㉑ Water Collector Tank ㉒ Brass Flange ㉓ N.B. Stand

㉔ Cover Plate (Brass).

Fig. A.2 : Schematic diagram of strip caster assembly

TUNDISH

LIQUID METAL

METAL STRIP

KNIFE EDGE
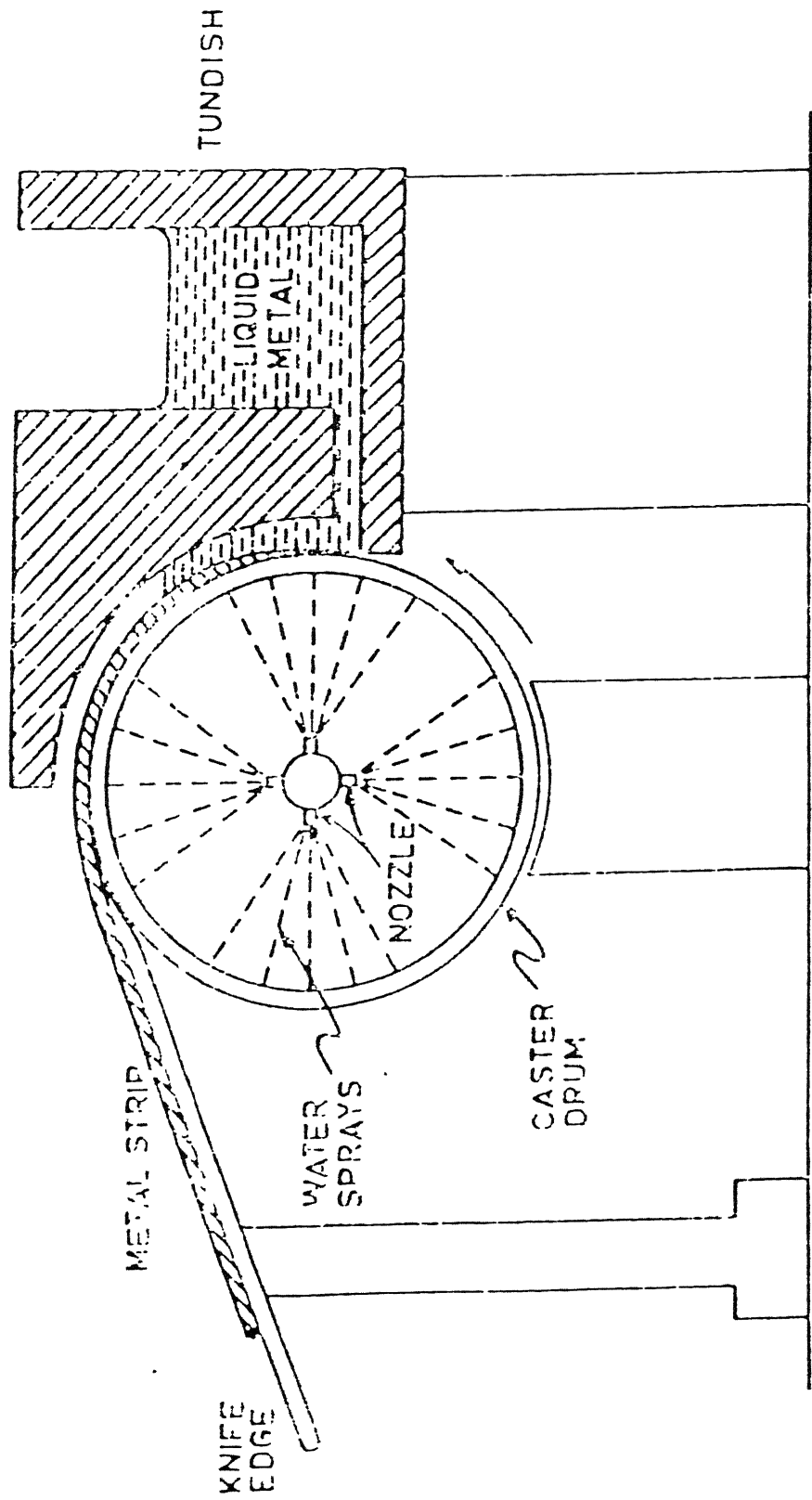
WATER SPRAYS

NOZZLE

CASTER DRUM

Fig. A.3 : Schematic diagram of caster drum assembly

The tundish is placed vary close to the caster drum without touching it to avoid scratching of the drum surface. At the same time it is ensured that the gap between the drum surface and the tundish is not large enough to lead to the leakage of the molten metal through it.

A.2 CASTER DRUM ASSEMBLY

The caster drum is made of high purity (99.99%) copper. It is a hollow cylinder with both ends open The total length is divided in two portions. one is the caster drum portion and the other one is water outlet portion, which is drilled with holes on its surface and provides an outlet for the cooling water. The inner surface of the drum is tapered towards the water exit as shown in Figs A 2 and A.3, to facilitate the easy flow of the cooling water. Both ends of the drum are fitted with brass flanges. One end of the caster drum is connected to the shaft of the microprocessor controlled stepper motor, which can vary the rotational speed of the caster drum between 1.5 RPM to 38 RPM. The other of the drum is connected to a water pipe line and also holds the water spray assembly inside the drum The caster drum and the water outlet portion of the drum are seated on the outer surface by a brass ring so that the exit water does not come in contact with the molten metal or the solidifying strip at any time. The caster drum is fitted with thermocouples at two points to measure the temperature continuously during the casting process.

A.3 WATER SPRAY SYSTEM

Water spray assembly consists of four nozzles placed at right angles to each other and in two rows as shown in Fig. A.3. The water spray covers almost the entire surface area of the caster drum to ensure its uniform cooling The spray nozzles are specially designed such that each nozzle generates a fully developed water cone with cone angle of about 70°. The nozzles are fitted through the manifolds on the horizontal stainless steel pipe, which passes through the brass flange on one of the open s of the caster drum This pipe remains stationary when the drum is rotating.

A.4 MECHANISM OF WORKING OF CASTER

The liquid metal with the reinforcing particles is held in the tundish at a particular temperature in which its level is always kept constant. The tundish has a rectangular opening at

the bottom of the side facing the caster drum. The molten metal continuously flows through this opening into the liquid pool contained in the annular space between the rotating caster drum and the tundish wall. The drum is water cooled to remove the heat of the molten metal. This rotating drum drags along with it a t of metal from the liquid metal pool, which on solidification forms a skin of solidified metal strip. The skin continues to grow as long as it is in contact with the molten metal in the pool. A knife-edge located on the other side of the caster drum peels off the solidified strip from the drum after it has drawn to its full thickness. Heat is continuously extracted from the solidifying strip through the caster drum by water spray nozzle located inside the hollow drum for a given liquid metal head in the tundish , strips of different thicknesses can be produced by varying the speed of the caster drum.

At the point of first contact between the caster drum and the liquid metal there is instantaneous solidification and skin of initial thickness is formed. This skin continuously grows during its journey through the liquid pool and it finally attains the desired thickness at the exit of the pool. The solidified strip remains in contact with the liquid pool over the annular position which in turn, is directly elated to the metal head in the tun-dish. The entire process can be divided into four distinct zones· (i) liquid in the tundish with nozzle, (ii) liquid metal pool, (iii) solidifying strip, (iv) water cooled caster drum.

The liquid pool is bounded by tun-dish walls and solidifying strip on the sides and the protruding tun-dish wall on the bottom side. The top side is free and open to the atmosphere. The molten metal from the pool is being continuously removed in the form of the solidifying strip and this loss of the metal in the pool is being replenished continuously by the flow of metal from the tun-dish. At steady state, the two rates, i. e the rate at which the strip is being produced and the rate at which metal is flowing in the pool, are equal such that the liquid metal level in the pool always remains constant. One surface of the solidifying strip is attached on to the caster drum whereas the other surface is interfaced with the molten metal in the liquid pool. It is assumed that the strip is firmly adhered to the caster drum and that there is no slip between the two. Also the strip is assumed to be moving at the same speed as that of the caster drum and the residence time of the solidifying strip in the liquid metal pool is directly related to the speed of rotation of the drum which, in turn, determines the final thickness attained by the strip as well as the production rate.

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